EVALUATION OF NEWLY DEVELOPED REINFORCED THERMOPLASTIC COMPOSITES

Contract N00019-81-C-0345

FINAL REPORT NO.

December 1982

C. H. Sheppard, E. E. House

Submitted to

NAVAL AIR SYSTEMS COMMAND
WASHINGTON, DC 20361

Submitted by

THE BOEING AEROSPACE COMPANY
ENGINEERING TECHNOLOGY ORGANIZATION
SEATTLE, WASHINGTON 98124

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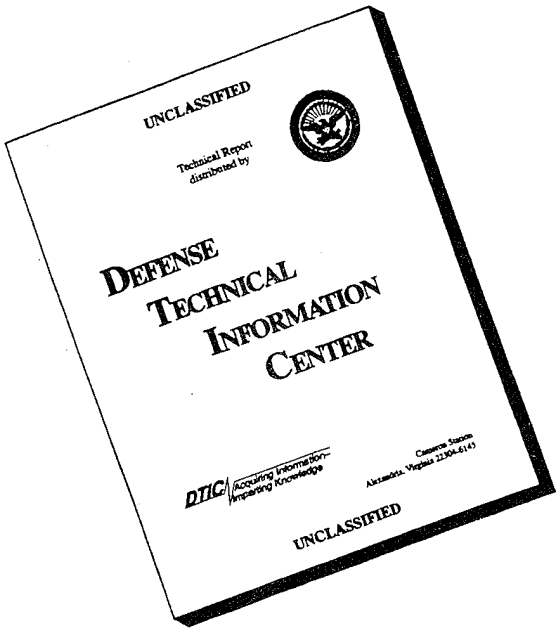

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FOREWORD

This report summarizes the work performed by the Boeing Aerospace Company from July 1, 1981 through December 1982, for the Naval Air Systems Command, United States Department of the Navy, under Contract N00019-81-C-0345, entitled "Evaluation of Newly Developed Reinforced Thermoplastic Composites." Mr. Maxwell Stander (AIR 5163D3) is the NASC project manager.

The program was conducted by the Engineering Technology Organization of the Boeing Aerospace Company, Seattle, Washington. Key personnel contributing to the program during this period were:

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SUMMARY

This document is the final program report describing work performed by the Boeing Aerospace Company (BAC) for the Naval Air Systems Command (NASC) under contract N00019-81-C-0345. The objectives of this program were to: 1) evaluate newly developed thermoplastics which have improved resistance to fluids and/or solvents frequently encountered in aircraft flight and maintenance operations, 2) evaluate high temperature resins as coupling agents for improving the interfacial attachment of thermoplastic materials and Kevlar in composite structure and 3) obtain a preliminary design base of graphite, Kevlar and graphite/Kevlar hybrid composites to be used in conjunction with the CH-46 work platform manufacturing technology program (ref. 1). These objectives were accomplished by: 1) screening five commercially available thermoplastic matrices by immersion in corrosive aircraft paint stripping agents, 2) screening the formability of four thermoplastic matrices by forming a 90° angle, 3) selecting the three most promising systems and obtaining design related data (i.e., tension and compression using graphite fabric as the reinforcement), 4) studying the effects on composite mechanical properties of applying several different high temperature polyimide resins as sizing to Kevlar reinforcements, and 5) obtaining design related data for graphite, Kevlar and graphite/Kevlar hybrid composites.

During the initial stages of the program the work consisted of the selection of five different thermoplastic resins. Selection criteria were established and reinforced composites were fabricated and evaluated. This resulted in the selection of norbornene terminated sulfone (NTS-20), polyphenylene sulfide (PPS) and polyetherether ketone (PEEK). The results of that characterization demonstrated that 1) PEEK possessed the best overall mechanical properties and solvent resistance on graphite fabric, 2) PPS possessed the best overall forming characteristics with good solvent resistance on graphite fabric and 3) NTS-20 possessed the best combination of mechanical properties (using graphite and Kevlar reinforcements) and formability in combination with acceptable solvent resistance.

The next phase of work consisted of the study of interfacial adhesion using NTS-20 and different high temperature sizings applied to Kevlar fabric reinforcement.

Composites were fabricated and then tested to obtain flexural and interlaminar shear strength data. The values indicated that the high temperature polyimide resins used as sizing on Kevlar fabric were compatible with the NTS-20 system, and yielded properties essentially equivalent to SOTA epoxy Kevlar composites.

Preliminary design development under the final phase of the work demonstrated the suitability of using NTS-20 thermoplastic matrix/graphite, Kevlar, and graphite/Kevlar hybrid composites. The specific data indicated that the NTS-20 system would perform similarly to SOTA epoxy systems on the CH-46 work platform.

1.0 INTRODUCTION

Over the past 10 years, government agencies, particularly NASC, and private industry have conducted studies with a goal of developing graphite/thermoplastic and Kevlar/thermoplastic composites suitable for use in aircraft structure. Through these years, the primary reason for these studies has been to reduce the original cost of advanced composites by taking advantage of the lower processing cost possible with thermoplastics as compared to the epoxy thermosets currently used.

During this time, the primary deficiency of thermoplastic matrices suitable for advanced composites use, has been degradation by certain aircraft fluids. This problem has been resolved. For example, an NASC/Boeing program (ref. 1) resulted in an invention in which polysulfone, the most suitable thermoplastic polymer available except for resistance to chlorinated solvents and some types of aircraft fluids, was chemically modified to make it resistant to those materials. The new polymer, designated NTS, is being further characterized and optimized on ongoing NASC/Boeing programs (refs. 2, 3).

In addition to the NTS polymer, commercially provided by Raylo Chemical Company, the aforementioned programs have acted as a stimulus for resin suppliers to develop other new polymers. For example: LaRC 2/TPI by Gulf or NASA Langley Research Center, a new resin, ULTEM by General Electric, a film version of polyphenylene sulfide developed by Phillips, and finally the Development of Modified Polyether-etherketone made by ICI.

The goal of this program was to evaluate newer thermoplastic resins with respect to their application in composite structures exposed to a typical military hardware environment. Areas of emphasis included the evaluation of matrix resistance to typical system fluids, development of improved processing methods, and an assessment of mechanical properties including toughness, fatigue, and creep. Concurrently, the fourth area of investigation will explore the resin/Kevlar fiber interface and methods of achieving improved resin/fiber bond.

The aim of the studies was to develop structural composite materials with the optimum combination of the following properties:

- o Fluid resistance
- o Ease of fabrication
- o Mechanical properties
- o Toughness and impact resistance
- o Creep and fatigue resistance

Specifically the objectives accomplished during this program were:

1. Evaluation of newly developed thermoplastic polymers as matrix materials for graphite and Kevlar reinforced composites.
2. Demonstration of the improved strain-to-failure and impact resistance of thermoplastic matrix composites as compared to graphite/epoxy composites.
3. Evaluation of the compressive fatigue and creep capabilities of graphite reinforced thermoplastic composites.
4. Evaluation methods for increasing the shear properties of Kevlar/thermoplastic composites.
5. Evaluation of NTS-20 composites for use the reference 4 program.

The program was conducted in five tasks to meet the program objectives:

- o Task I—Resin Characterization and Fluid Screening
- o Task II—Process Optimization
- o Task III—Laminate Properties and Fluid Resistance
- o Task IV—Kevlar/Thermoplastic Interfacial Evaluation
- o Task V—Preliminary Design Data Using NTS-20 Thermoplastic Resin

2.0 TECHNICAL DISCUSSION

2.1 TASK I—RESIN CHARACTERIZATION AND FLUID SCREENING

During this task, candidate resin systems were selected and screened by means of a series of chemical and physical characterization tests and fluid immersions. Tests for the NTS-20 system included gel permeation chromatography (GPC) for average molecular weight, number average molecular weight, and polydispersitive factor; all the systems were further evaluated by scanning calorimetry (DSC), which indicates glass transition temperature, infrared (IR) analysis, and dynamic mechanical analysis (DMA). Results of these tests, taken collectively, were used to help in the selection of conditions in Task II, provide a guide for high-temperature capability, and establish a baseline set of fingerprint characteristics for each material evaluated.

In addition to the above, screening tests using graphite reinforced composites were conducted to judge, qualitatively, the fluid resistance of each material. In this test, both stressed and unstressed composite samples were placed in contact with key system fluids. After a standard exposure time (92 hours), samples were examined under magnification for crazing, evidence of solvation, or other degradation. This screening eliminated clearly unsuitable materials. Materials passing this screening were evaluated in Task II—Fluid Exposure Tests.

2.2 TASK II—PROCESS OPTIMIZATION

Material evaluated in Task I underwent additional tests to determine optimum processing conditions. These tests included both the conditions for consolidation of prepreg into flat stock and the process parameters used in postforming.

Sheet consolidation variables studied included temperature and pressure levels, preheat time, and, where applicable, postcure conditions. The test matrix for each material was designed in advance to allow the isolation of effects by single variables, and thus optimize each process in the least number of trials. Evaluation was based on the resulting mechanical properties, including flexural strength and modulus and short beam shear, and on nondestructive tests (through-transmission ultrasonic).

Postforming tests were aimed at identifying both the formability of the matrix material and the most rapid forming process consistent with maintaining mechanical properties. Simple shapes were formed using matched die stamping techniques. Subsequent tests involved examination for fiber damage and limited mechanical property testing.

Using the processes developed, the fluid resistance of the materials was evaluated in conventional maintenance operation solvents and paint strippers.

2.3 TASK III—LAMINATE PROPERTIES AND FLUID RESISTANCE

Task III tests included the static design properties of tension and compression strength and stiffness and strain-to-failure. Since one of the key advantages of thermoplastic matrix composites is toughness, additional tests were designed to measure that characteristic.

Basic tension and compression properties were run on graphite fabric reinforced panels. Elevated-temperature tests were conducted at temperatures appropriate to the resin system—the standard test temperature was 300°F except for PPS and PEEK which were tested at 200 and 350°F, respectively.

This phase included stressed and unstressed exposures to typical aircraft fluids. Flexural specimens prepared from quasi isotropic layups (i.e. 0,90,$\pm$45) were placed under load for stressed exposures, held in contact with fluid for 30 days at room temperature, and tested to failure.

In addition to basic mechanical tests and fluid exposures, source-related testing included measures of toughness and fatigue resistance.

A drop-weight impact test, (ref. 5) studied at Boeing and found to be a sensitive indicator of resin system properties, was the technique used to measure toughness. In this test, a 6 x 6-inch panel was impacted at its center by a weighed indenter dropped from a specified height. Plots of load versus deflection were obtained and used to establish the incipient damage point (P_I) and peak load sustained (P_F) during the test. Substantial data on epoxy-based systems is available for comparison. G_{IC} testing was conducted on constant-thickness specimens.

Limited fatigue testing was performed on only the NTS-20 and PPS composite systems. Since compressive fatigue loading appears to result in greater life reduction than tension-tension loading (ref. 6), this test was selected.

2.4 TASK IV—KEVLAR/THERMOPLASTIC INTERFACE EVALUATION

Concurrent with the work conducted in Tasks I, II, and III, studies of the fiber/resin bond in Kevlar composites were conducted and added to the information generated on NASC Contract N00019-80-C-0365 (ref. 2). Unsized Kevlar fabric was pretreated by addition of a chemical finish. A thermosetting high temperature polyimide resin was deposited from a dilute solution onto Kevlar fabric (unsized, dried to constant weight) then cured.

From the initial selection of candidate resins, the NTS-20 appeared to be the most promising and was used in this study. It was proposed to also include both a melt-processing and a reactive resin. The treated fabric was used to prepare laminates for flexural strength testing. Following testing to failure, the fabric surfaces were examined by photographing polished sections. Using the comparative flexural strengths and visual evidence, the various fiber treatments were ranked for effectiveness.

2.5 TASK V—PRELIMINARY DESIGN DATA USING NTS-20X THERMOPLASTIC RESIN

The objective of this task was to obtain sufficient design allowable data to insure that the NTS-20/graphite/Kevlar structure designed for the work platform of the CH-46 helicopter (ref. 7) would meet design goals. Specifically, this task evaluated the physical and mechanical properties of graphite/NTS-20X and/or graphite hybrid/NTS-20X composites as required for the final design configuration of the CH-46 hinged work platform studies in NASC/Vertol programs. The materials for these studies were provided in limited quantities by Boeing Vertol at no cost to the program.

3.0 TECHNICAL ACCOMPLISHMENTS

This program consisted of experimental studies structured to select and evaluate thermoplastic resins with regard to their processibility and sensitivity to typical aircraft fluids. Based on this evaluation, the most promising candidates were selected and additional tests conducted. The program was divided into five separate tasks:

Task I—Resin Characterization and Fluid Screening
Task II—Process Optimization
Task III—Laminate Properties and Fluid Resistance
Task IV—Kevlar/Thermoplastic Interfacial Evaluation
Task V—Preliminary Design Data Using NTS-20 Thermoplastic Resin

3.1 TASK I—RESIN CHARACTERIZATION AND FLUID SCREENING

The objective of this task was to evaluate various thermoplastic materials with respect to their resistance in typical aircraft paint stripping agents, and to compare the performance with that of epoxy materials being currently used in aircraft structure. The task was accomplished in various phases which are summarized below:

- o Material Selection
- o Preliminary Composite Evaluation
- o Environmental Exposure
- o Selection for Additional Evaluation

A more detailed discussion of the experimental work is presented in the following sections.

3.1.1 MATERIAL SELECTION

The basic criteria used in the selection of candidate materials were that the materials 1) be processible into structure using thermoplastic forming techniques (i.e., be reformable, after having been consolidated, with a full compliment of properties), 2) to be relatively insoluble in currently used fluids and/or solvents available to Navy flight and maintenance operations and 3) be commercially or near to being commercially available. The resin selection was made using data obtained from previous Navy programs, governmental reports, professional papers and material

suppliers. Five resins were selected for evaluation under this effort and are given below:

1. Imide Capped Polysulfone (NTS-20)
2. Ryton Polyphenylene Sulfide, film (PPS)
3. Polyetherether Ketone (PEEK)
4. LaRC TPI
5. Ultem Polyetherimide

A brief discussion follows pertaining to the selected resins.

3.1.1.1 NTS-20

Work on previous NASC contracts (ref. 1, 2) demonstrated that this imide terminated polysulfone possessed good potential of meeting the Navy's requirements for thermoplastic resins. However, all material used was made in the BAC polymer laboratory in small batches (i.e., one half pound). One of the requirements of this program was to use commercially manufactured material (para. 3.1.1) hence the NTS 20 used on this program was procured from Raylo Chemical Company, Edmonton, Alberta Canada. The resin was first chemically characterized using procedures developed on contract N00019-80-C-0609, then impregnated onto graphite fabric, consolidated into composite panels and the mechanical properties determined. The NTS-20 resin was then supplied to U.S. Polymeric and prepregged using 3K-70-PW/T300 graphite fabric (see Table I). Data thus generated demonstrated the NTS-20 polymer, when made into reinforced composites, possessed properties similar to SOTA epoxies (i.e., Hercules AS1/3501-6A).

3.1.1.2 PPS

The film form of polyphenylene sulfide produced by Phillips Petroleum Company was evaluated on Navy Contract N00019-80-C-0365 with somewhat promising results. Work on that program amply demonstrated the material to possess excellent chemical resistance as well as satisfactory processability. The data generated by BAC corroborated the previous work published by the University of Dayton research institute (ref. 6).

TABLE I

PROPERTY SUMMARY TASK I STUDIES

MATERIAL	Flex Stress ksi RT	Flex Stress ksi 300°	Flex Mod Msi RT	Flex Mod Msi 300°	ILS ksi RT	ILS ksi 300°
PPS	69.2	48.5 1/	9.3	7.7 1/	6.9	4.2 2
LaRC-2/TPI	87.8	94.6	10.2	9.8	8.4	7.1
Ultem	57.3	52.5	10.6	9.7	6.4	4.0
NTS-20	107.7	71.0	11.0	12.0	9.2	5.6
PEEK	79.0	65.8	7.8	8.0	10.8	7.2
SOTA Epoxy 2/	96.5	---	8.7	---	10.0	---

1/ Test temperature 200°F. Values obtained at 300°F were 18.0 ksi flexural and 2.0 ksi shear and were considered inadequate.

2/ Hercules AS1/3501-6A only tested at room temperature.

3.1.1.3 PEEK

Cast films of polyetheretherketone (PEEK) were also evaluated on Navy Contract N00019-80-C-0365 with excellent results. The only problem encountered on that program was the availability of sufficient quantities of the material to perform the required tests. In conversations with the representatives of ICI, the resin supplier, BAC was assured that a sufficient quantity of film would be made available for the requirements of the program.

3.1.1.4 LaRC 2/TPI

The material is a pseudo thermoplastic polyimide system, developed by Langley Research Center (LaRC) for use in high temperature adhesive systems. Tentative evaluation by LaRC indicated that the material exhibited good potential for making graphite reinforced composites. The processing conditions of the system were unknown but essentially consisted of taking medium weight oligomers in polar solvents, solvent impregnating the reinforcement (e.g., graphite or Kevlar) and then autoclave curing. The curing conditions appear to be similar to those being used with the NTS materials. The major properties making this type of material attractive would be its compressive properties and potential for good fatigue life. The major deficiencies of this type of system are the condensation cure mechanism of polymerization, and resin cost. Enough material was obtained from LaRC to accomplish sufficient testing to determine if the material would meet the criteria established in para. 3.1.1.

3.1.1.5 Ultem

This material is a polyetherimide resin similar to the NTS-20 except that the system is linear in nature, compared to the crosslinked NTS-20 materials. Another major difference is the molecular weight of the Ultem, which is considered high versus the oligomeric weights of the NTS materials. This high molecular weight presents somewhat of a problem of making prepreg from graphite and/or Kevlar reinforcements. The ability of the resin to wet the reinforcing fibers is unknown. The resin properties obtained from product literature indicate the mechanical and/or physical properties to be somewhat better than P1700. However, exhaustive testing by the manufacturer, G.E., in various solvents and/or fluids indicated that the material was susceptible to environmental stress cracking (ESC) by chloroform, methylene chloride, phenols and some other chlorinated solvents.

3.1.2 CHEMICAL CHARACTERIZATION OF CANDIDATE SYSTEMS

This phase of the program was conducted to obtain some chemical characteristics of the candidate materials. The purpose of this effort was twofold: 1) to obtain baseline data for quality control purposes and 2) to aid in determining key processing conditions. A more detailed discussion concerning each system follows:

3.1.2.1 NTS-20

The NTS-20 material used during the course of the program was produced at Raylo Chemical Company in a 100 lb pilot plant batch. Samples of the resin were analyzed at BAC Quality Control labs using the following tests: 1) High Pressure Liquid Chromatography (HPLC) Gel Permeation Chromatography mode (GPC), 2) elemental analysis and 3) infrared spectroscopy. The NTS-20 material obtained was very similar in average molecular weight, average number molecular weight and polydispersitive factor to the standard materials made in the BAC polymer laboratories. In addition the elemental analysis and infrared spectrum compared favorably with BAC standards (see Table II).

3.1.2.2 PPS

The PPS film received from Phillips to be used on this program, was chemically characterized by infrared spectroscopy, Dynamic Mechanical Analysis and DSC (Figures 1 thru 3). No surprises were encountered, and the test results were used as quality control data.

3.1.2.3 PEEK

The PEEK film was chemically characterized by infrared spectroscopy DMA and DSC (Figures 4, 5, and 6). No surprises were encountered and the test results were used for determining processes and as quality control data.

TABLE II

SUMMARY OF DATA SECOND BATCH NTS-20X
RESIN FROM RAYLO CHEMICAL

GPC Analysis	Avg Mol WT	No. Avg Mol WT	Polydispersitive Factor
Raylo Batch			
Laboratory Washed Sample	23,313	7,378	3.1
Plant Washed Sample	21,169	4,705	4.5
BAC Standard	19,920	4,480	4.1
Typical Laboratory Sample	22,458	6,533	3.4

IR Analysis	Imide [1] Aromatic-O-Aromatic	Carbonyl Aromatic-O-Aromatic	Sulfone Aromatic-O-Aromatic
Raylo 100 lb Batch	15.2	7.5	73.9
BAC Standard	11.8	4.5	82.1

[1] Ratio of Wavenumbers characteristic of absorption band

3.1.2.4 LaRC 2/TPI

NASA Langley (Dr. Terry St. Clair) provided a gallon of LaRC 2/TPI varnish to BAC for evaluation. A limited amount of chemical characterization was conducted using LaRC 2. The data obtained included the infrared spectrum and DSC. Additional characterization was suspended because of the inability to reform the material in Task II studies (see Figures 7 and 8 for data).

3.1.2.5 Ultem

A limited amount of chemical characterization was conducted using Ultem. Since the material was dropped from further consideration, no additional characterization was contemplated.

3.1.3 PRELIMINARY COMPOSITE EVALUATION

This phase of the program was to fabricate sufficient composite using the five candidate materials selected in para. 3.1.1. Data obtained from the resin supplier, literature and the chemical characterization was used to obtain the processing conditions used in this study. A more detailed description of the processing conditions is given in Appendix A. The conclusions derived from the solvent screening tests for each system are given below.

3.1.3.1 NTS-20/Graphite Composite Evaluation

The 100 pounds of the commercially made NTS (manufactured by Raylo Chemical Co.) was chemically characterized (para. 3.1.2.1) and supplied to U.S. Polymeric for impregnation on 3K-70-PW/T300 graphite fabric. A sufficient quantity of graphite composite was then fabricated to evaluate its sensitivity to $MeCl_2$ and other paint stripping solutions (see Table III). One set was placed in $MeCl_2$ and the second set in a solution of 1 part 90% phenol and 3.5 parts $MeCl_2$. This solution was developed by personnel at Naval Air Development Center to simulate paint strippers now in use in the Navy.

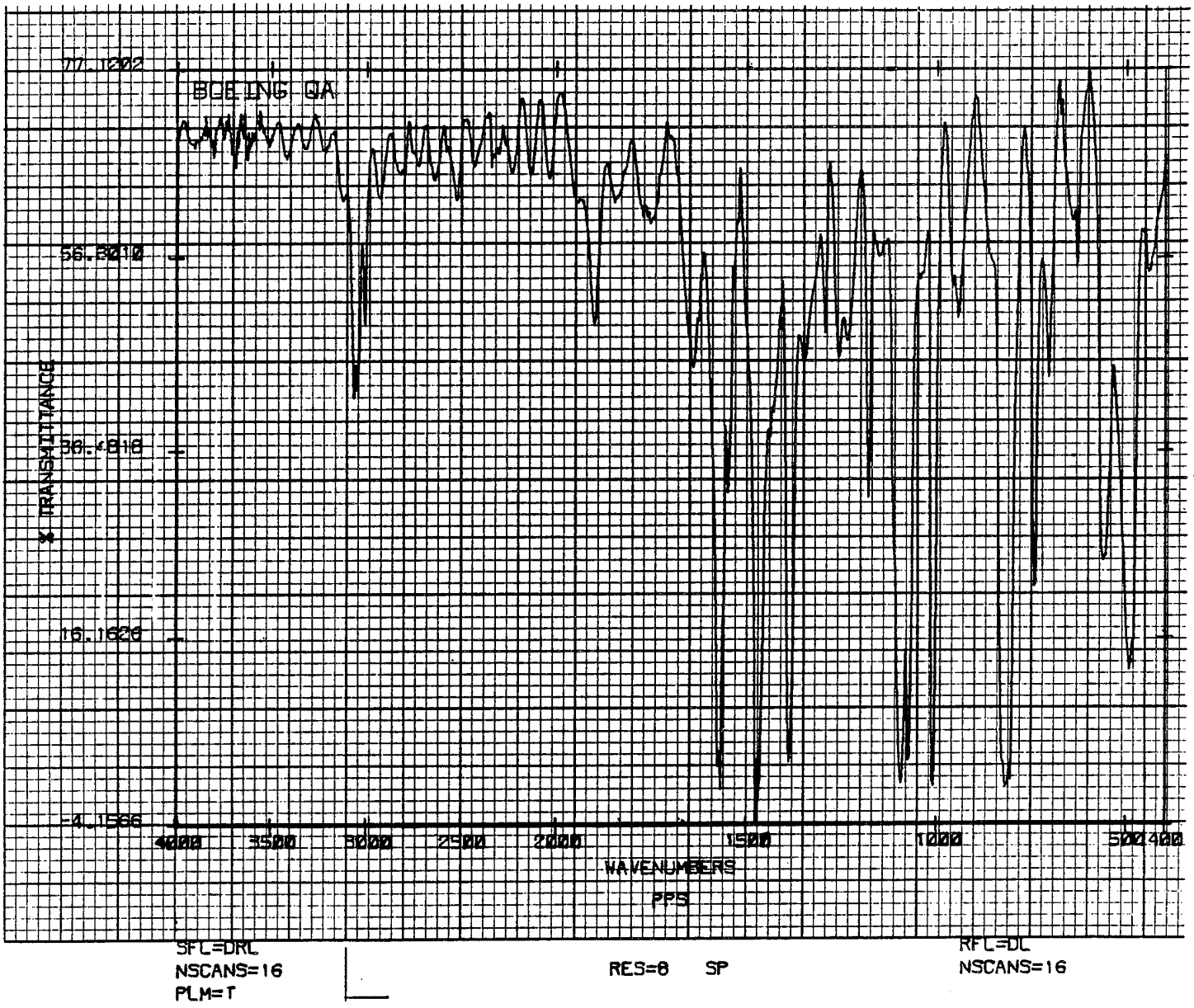


Figure 1

Infrared Spectrogram PPS Film

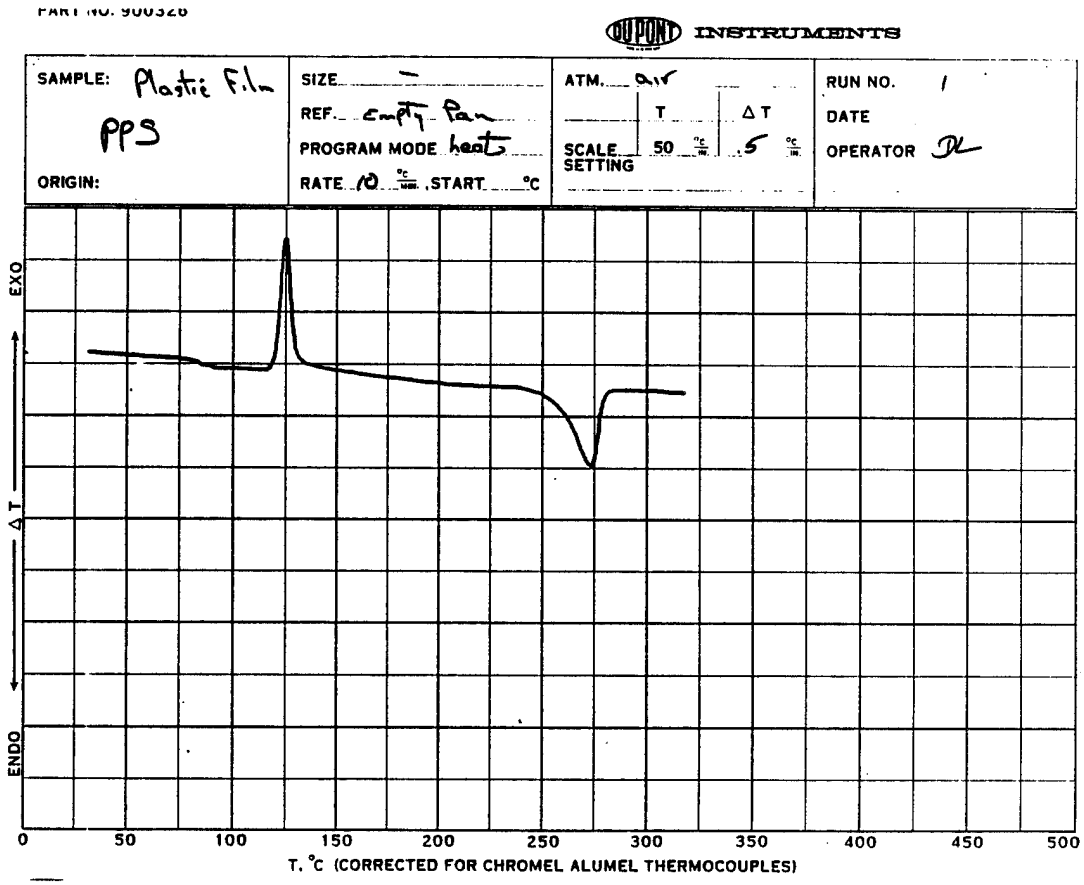


Figure 2

DSC Scan PPS Film

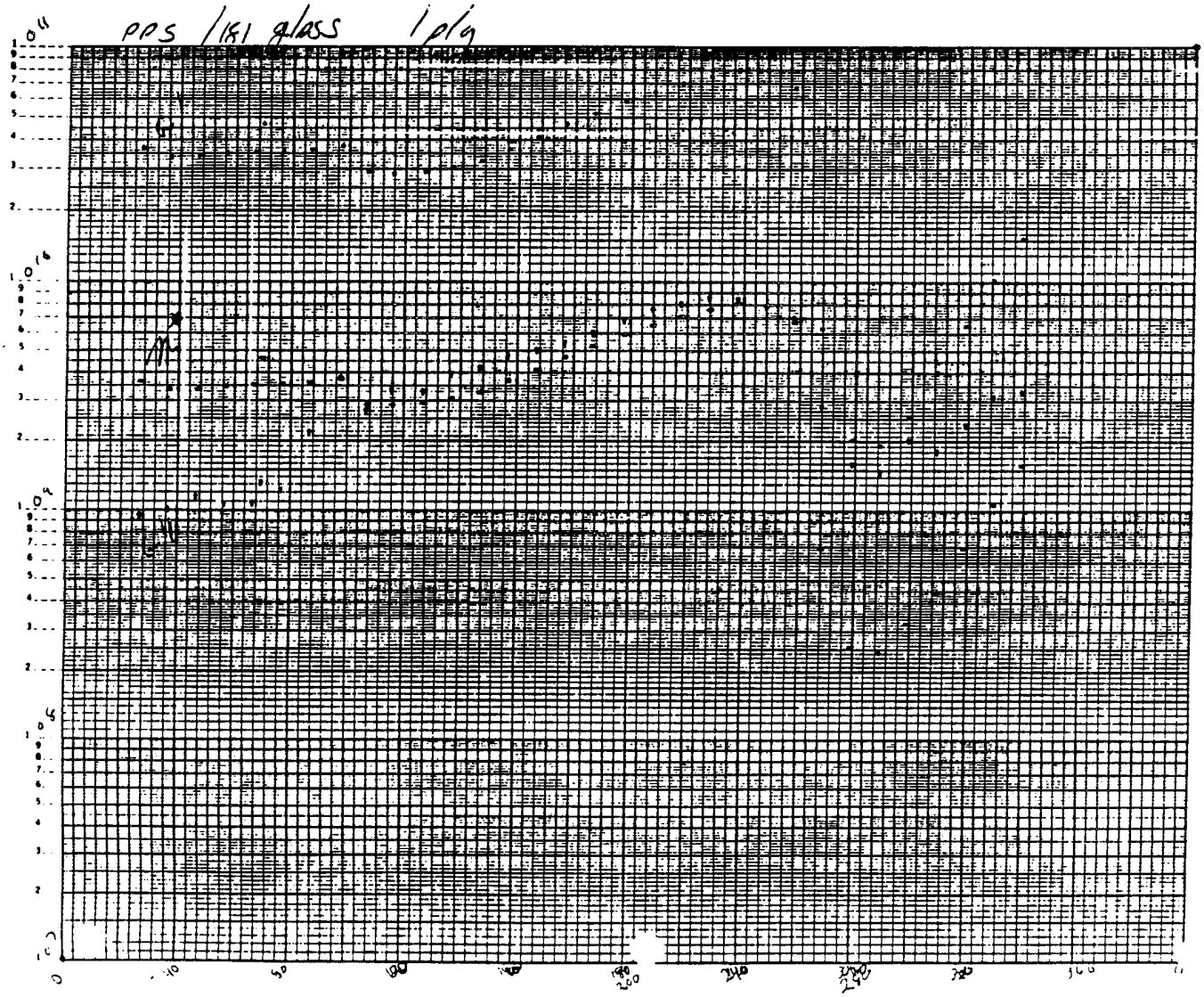


Figure 3

Dynamic Mechanical Analysis PPS

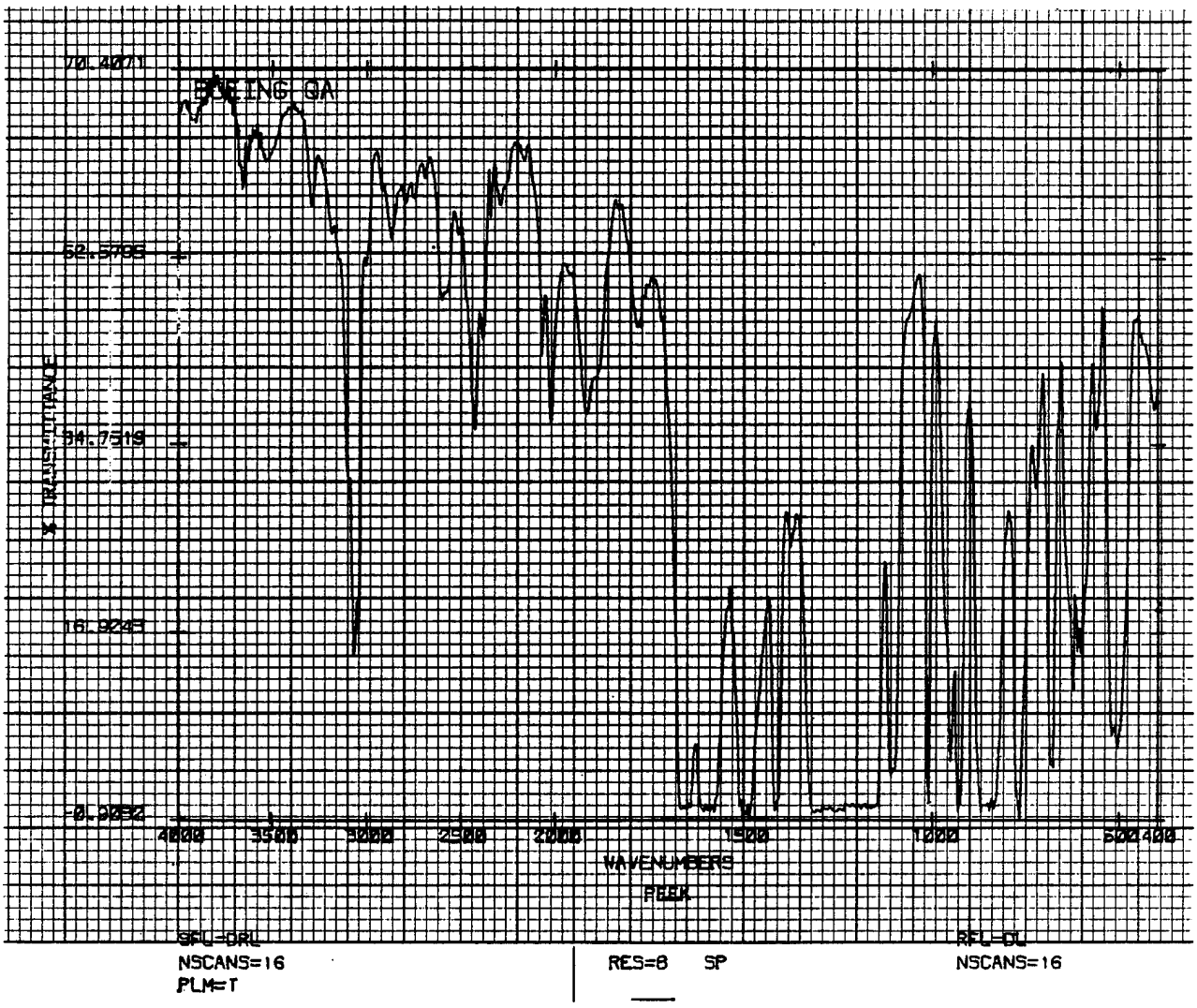


Figure 4

Infrared Spectrum PEEK Film

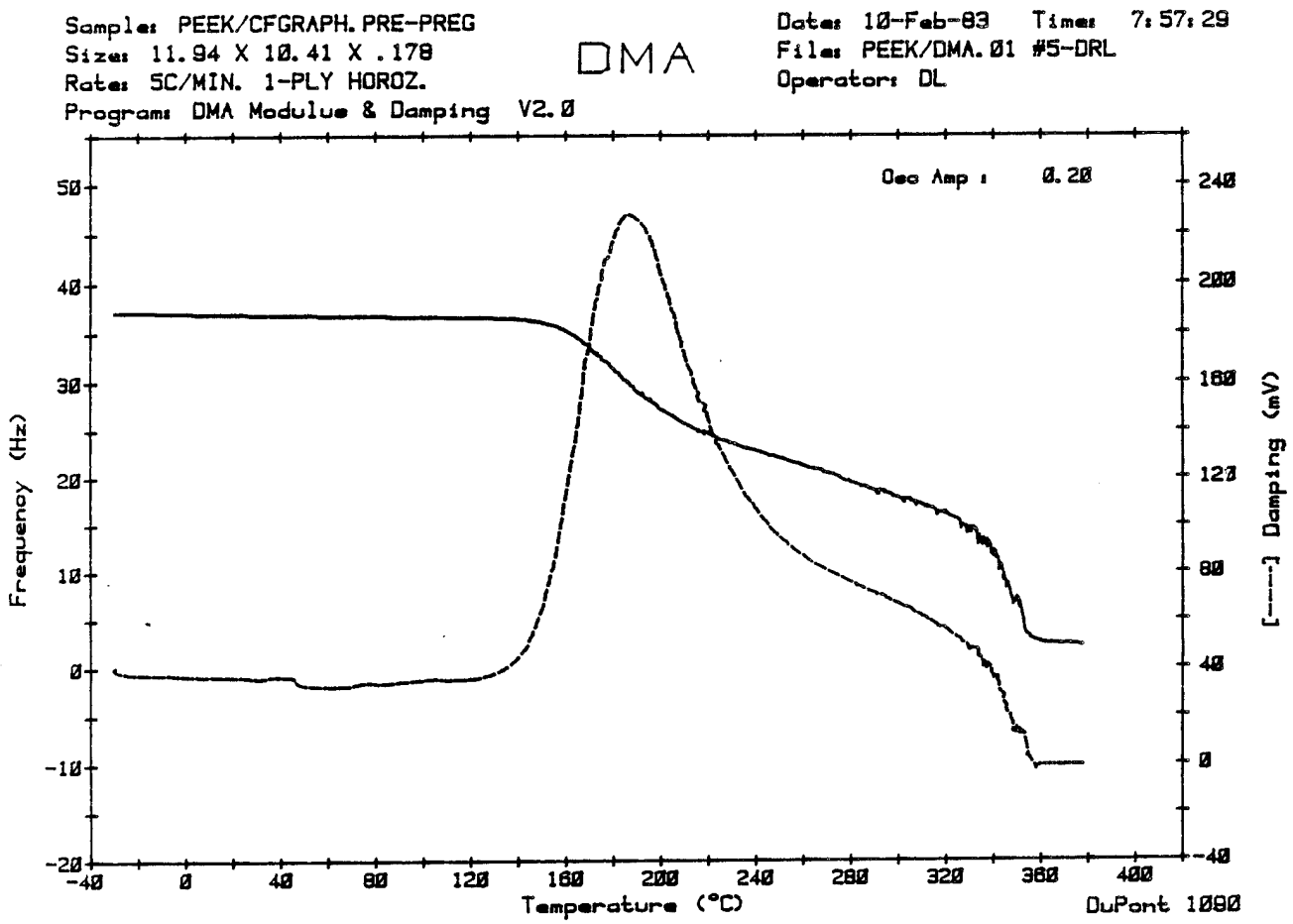


Figure 5

Dynamic Mechanical Analysis PEEK

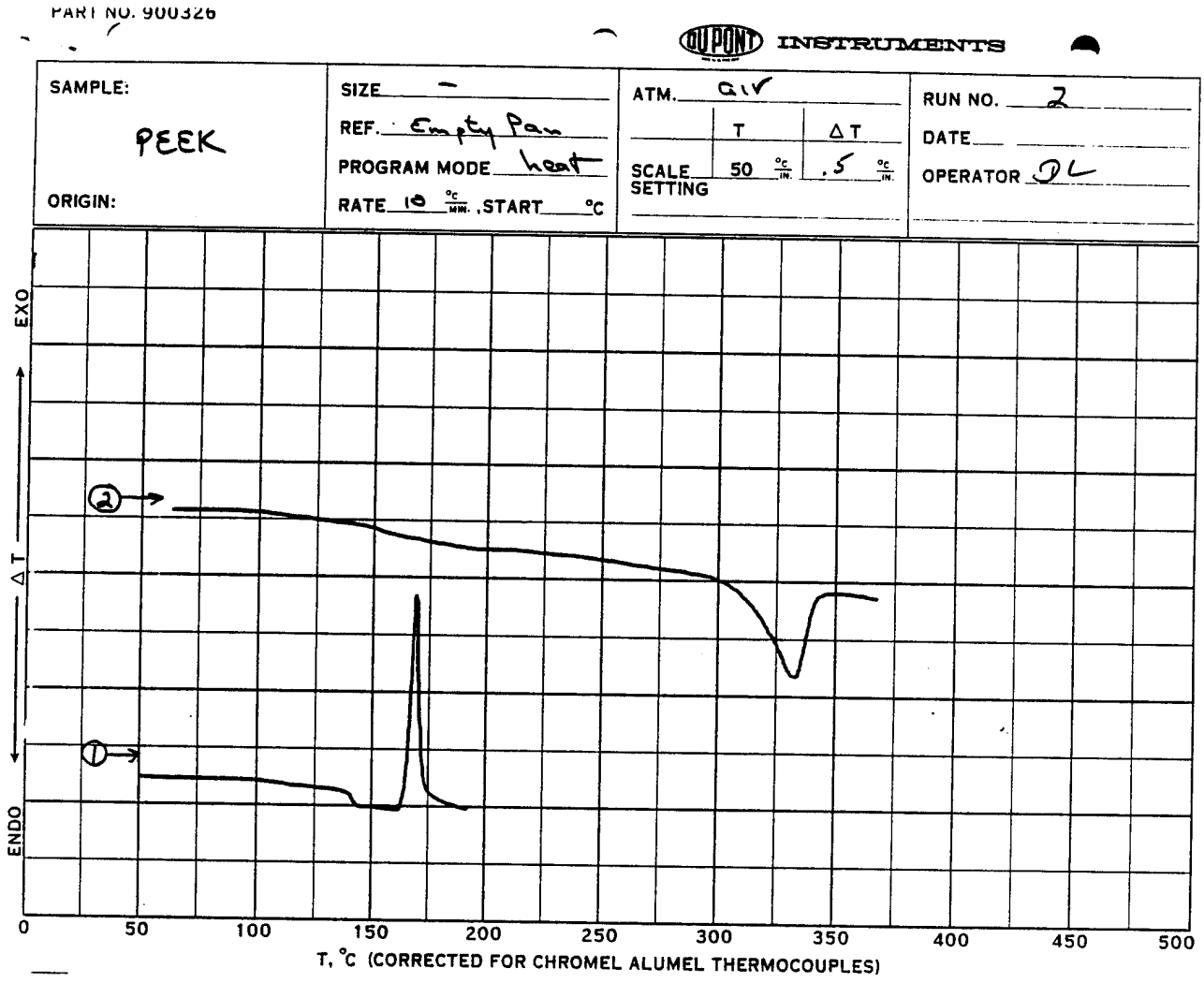


Figure 6

DSC Scan PEEK

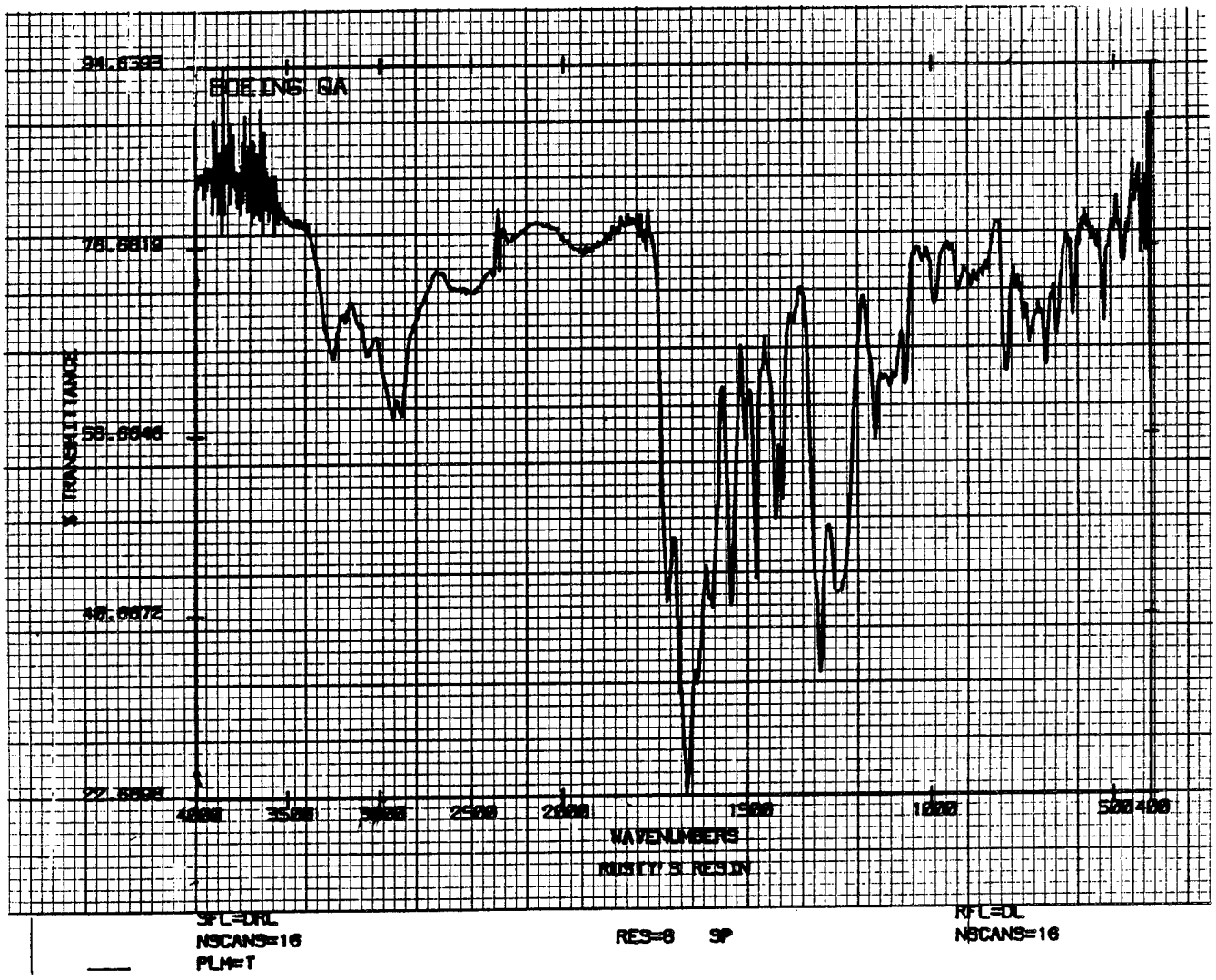


Figure 7

Infrared Spectrum LaRC 2/TPI

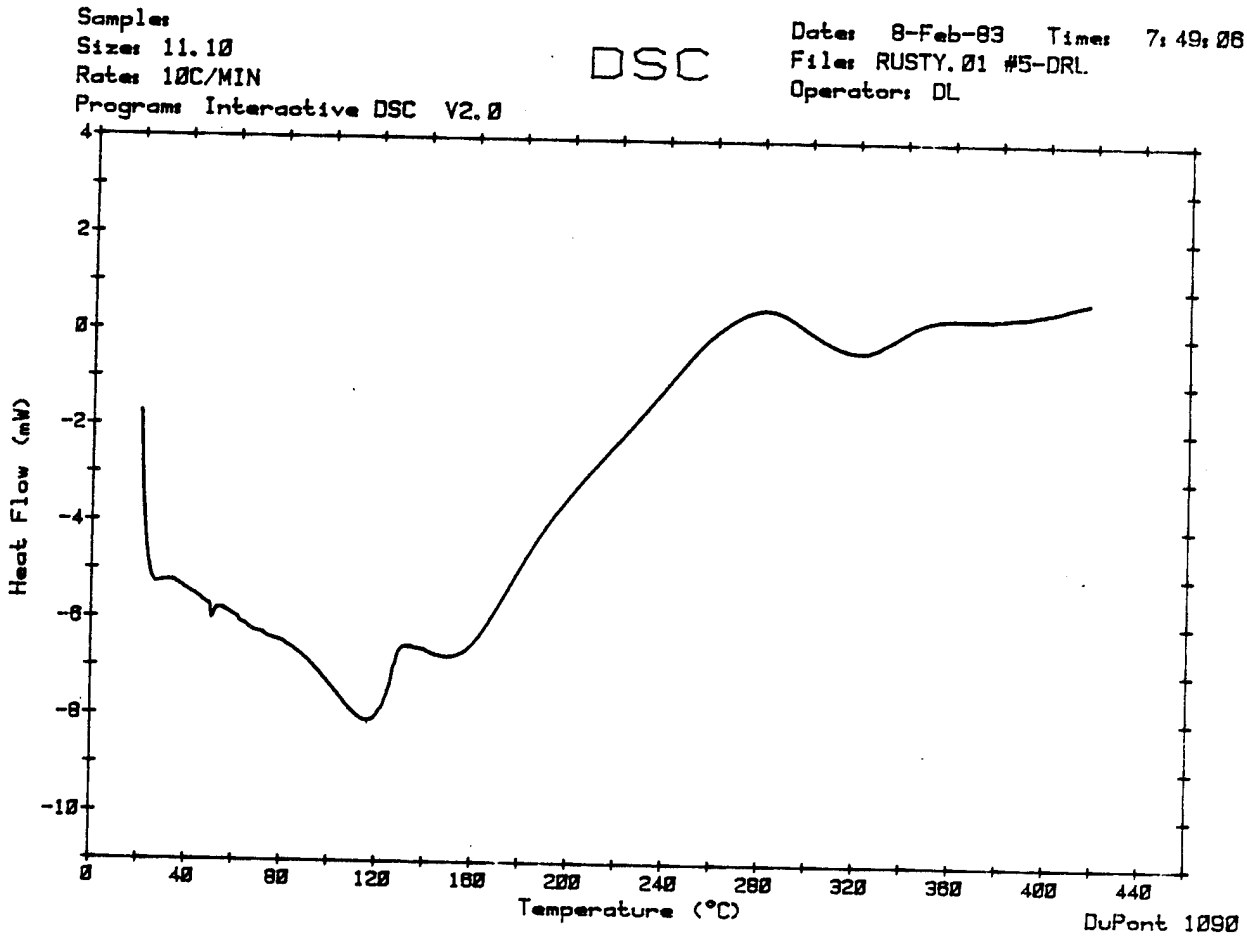


Figure 8

DSC LaRC 2/TPI

TABLE III

SUMMARY MECHANICAL PROPERTIES GRAPHITE COMPOSITES
USING RAYLO'S NTS-20 RESIN

Properties	Laboratory Washed	Plant Washed
Flexural Ult ksi		
RT	88.4	95.2
300°F	65.0	70.0
Flexural Modulus		
RT	7.4	7.7
300°F	7.6	7.9
Interlaminar Shear		
RT	8.0	9.5
300°F	6.6	5.6
Resin Content	35.0	32.1
Specific Gravity	1.53	1.55
Fiber Volume	56.5	59.1
Void Volume	1	1

The stressed specimens remained immersed in the test solutions for 90 hours, and then were tested to failure (Table IV). The results were somewhat disappointing in that the specimens from set two completely delaminated. The results of the $MeCl_2$ tests were satisfactory and duplicated tests conducted on the previous NASC contract (ref. 1)

3.1.3.2 PPS/Graphite Composite Evaluation

Composites were fabricated using T300 plain weave fabric style 3K-70-P with epoxy compatible sizing (UC 309) and PPS film. Conditions used in the consolidation cycle were developed on NASC Contract N00019-80-C-0365. The consolidation cycle is summarized below.

To prepare components at 37 weight percent resin, 11 plies of graphite fabric were interleaved with a sufficient amount of PPS film, and the assembly was vacuum bagged and heated for 60 minutes in an autoclave at 200 psi and 600°F.

In the first series of tests, the flexural stress, flexural modulus and interlaminar shear values were obtained (Table I). To determine the resistance to simulated paint stripping agents, flexural specimens were mounted in test fixtures, loaded to 40% and tested in the same manner at the NTS-20 specimens (para. 3.1.3.1). The results in both solutions showed the PPS to be relatively insensitive, retaining a significant percentage of its original strength. For comparative purposes, the same test was conducted using a conventional 350°F curing epoxy system. The epoxy system showed very little degradation in either solvent.

Based on these data a series of forming tests was conducted in Task II using a 90° angle matched die tool capable of simulating vacuum forming.

TABLE IV

SUMMARY OF DATA

STRESSED AGED IN SIMULATED PAINT STRIPPERS 90 HOURS

Property	PEEK	SOTA Epoxy 1	SOTA Epoxy 2	Modified Epoxy 1	Modified Epoxy 2	LaRC TPI	PPS	NTS-20
Flex ksi								
RT	95.0	96.5	108.6	83.7	87.3	87.8	76.6	107.7
Msi								
RT	10.1	8.7	8.0	7.2	6.2	9.2	9.3	9.5
Stress Flex $MeCl_2$								
ksi								
RT	106.6	105.3	104.5	76.1	65.1	83.0	49.8	36.2
Msi								
RT	10.6	9.0	7.7	7.2	5.4	9.0	7.9	9.0
Stress Flex[1/] $MeCl_2$/phenol								
ksi								
RT	114.4	113.5	116.7	64.5	66.7	80.4	48.8	delam
Msi								
RT	10.8	9.0	7.0	7.0	7.0	8.5	8.1	delam
ILS								
ksi								
RT	10.8	10.0	10.7	8.0	8.0	8.4	6.4	9.2

1/ 3.5 parts $MeCl_2$

1.0 parts 90% phenol

3.1.3.3 PEEK/Graphite Composite Evaluation

Sufficient PEEK film (produced by ICI) was laminated into graphite composites, and the composites were evaluated by obtaining flexural and interlaminar shear properties (Table I). Preliminary screening using the PEEK film demonstrated the film to be insoluble in methylene chloride; even when the film was subjected to stressed tests with methylene chloride no effect could be detected. Sufficient PEEK film was interleaved with T300 plain weave fabric style 3K-70-P to make a composite 6" x 6" by approximately .080 inches. The assembly was vacuum bagged and placed into a press for consolidation. The consolidation cycle involved heating the assembly, under vacuum pressure, to 750°F then applying 1000 psi and holding for 60 minutes before cooling under pressure to ambient conditions.

As with the other systems the flexural stress, flexural modulus and interlaminar shear values were obtained. Then some flexural specimens were mounted in test fixtures and loaded to 40% of ultimate and one set immersed in $MeCl_2$ and the other in $MeCl_2$/phenol solution for 90 hours and then tested to failure (Table IV). The PEEK did not lose strength; it compares favorably with the SOTA epoxy and is better than PPS and the modified epoxy. The material was then evaluated in Task II for its formability.

3.1.3.4 LaRC 2/Graphite Composite Evaluation

Composites were fabricated using style 3K-70-P T300 fabric and LaRC 2. The fabric was impregnated with the LaRC 2 resin from a diglyme solution. The impregnated fabric was dried at ambient conditions and then cut and stacked into 10 ply laminates. The preplied laminate was vacuum bagged using fiberglass as a surface bleeder separated from the prepreg by porous teflon coated fabric. The vacuum bagged assembly was then autoclaved cured using the following cycle.

> Under a vacuum and 200 psi pressure, the assembly was heated to 400°F and held 60 minutes. It was then heated to 650°F and held 2 hours before cooling to ambient conditions.

The graphite composite mechanical properties (see Table I) and solvent stability in methylene chloride and in phenol/$MeCl_2$ were determined. Both were excellent (see Table IV). Based on these data a series of forming tests were conducted using a 90° angle to match the mold and a platen press in Task II.

3.1.3.5 Ultem/Graphite Composite Studies

Composites were fabricated using style 3K-70-P T300 fabric and Ultem. The fabric was impregnated with the resin from a methylene chloride solution. Some difficulty was experienced in quickly dissolving the resin to prepare the prepregging solution. Composites were fabricated using the following consolidation cycle:

> 11 plies of Ultem/graphite prepreg were laid up and vacuum bagged. The vacuum bagged assembly was introduced into an autoclave and heated at 200 psi, 600°F for 60 minutes.

In the first series of tests the flexural stress, flexural modulus and interlaminar shear values were obtained (Table I). Reasonable properties were obtained; however, when samples of the Ultem/graphite composite were immersed in methylene chloride or phenol/$MeCl_2$, the composite disintegrated. Hence, additional studies were not conducted using the Ultem system.

3.2 TASK II—PROCESS OPTIMIZATION

The objective of this task was twofold: first to develop processing conditions for consolidation of the thermoplastic materials into flat composites, and second to develop the processing conditions necessary to convert the flat composites into a 90° angle without damaging the reinforcing fibers. This task was generally accomplished in conjunction with Task I (para. 3.1), but for reporting purposes will be discussed separately. For convenience of reporting, the discussions given below will be separated by system.

3.2.1 GRAPHITE/NTS-20 PROCESSING AND FORMING STUDIES

Sufficient graphite/NTS-20 composites were fabricated using Raylo-supplied NTS-20 resin and U.S. Polymeric prepregged 3K-70-PW/T300 graphite/NTS-20 broadgoods. The studies were conducted in two phases: cure cycle development, and graphite composite forming studies.

3.2.1.1 NTS-20 Process Development

On previous NASC programs sufficient data was developed to guide this study. Graphite composite panels were fabricated as described in Appendix A. Three separate cure cycles were evaluated: 1) 4 hours at 650°F 200 psi, 2) 6 hours at 650°F 200 psi, and 3) 8 hours at 650°F. The resulting composites were machined into test coupons and tested. The results, reported in Table V, indicate that 6 hours at 650°F was adequate. This generally corresponds to the processing cycle used on previous programs (ref. 1, 2).

3.2.1.2 NTS-20 Forming Studies

Based on these data, a series of panels was prepared to study the effects of the above cure cycle on the forming characteristics. The forming tests were conducted as for the PPS and LaRC 2 systems, using a 90° angle match die mold. In each case, the mold and composite were heated to a predetermined temperature in the press. The composite was then placed within the mold and the mold closed using 200 psi pressure. The press was then cooled and the part removed from the mold. The formed angle was polished and microscopically examined to determine the effects of the forming operation (see Figures 9 thru 13). The results of the test indicate that under the conditions used, the crosslinking of the NTS-20 had some effect on the formability; in some cases there was evidence of fiber breakage. However, this test did amply demonstrate that using the proper forming conditions the NTS system can be formed into shapes and meets the original requirements of the program (see para. 3.1.1). For a summary of the forming conditions see Table VI.

TABLE V

SUMMARY NTS-20/GRAPHITE COMPOSITE

CURING CYCLE STUDY

	4 Hrs 650°F	6 Hrs 650°	8 hrs 650°
Flexural ult (ksi)			
RT	73.5	93.0	90.1
300°F	42.5	56.3	53.1
Flexural Mod (Msi)			
RT	7.7	7.2	7.5
300°F	6.2	7.4	7.3
Interlaminar Shear			
RT	6.7	6.5	6.8
300°F	3.5	5.1	5.2
Resin Content %	34.7	33.0	33.8
Specific Gravity g/cc	1.53	1.55	1.54
Void Volume %	2.3	1	1
Fiber Volume %	57	59	58

(1) Panel fabricated but not tested.

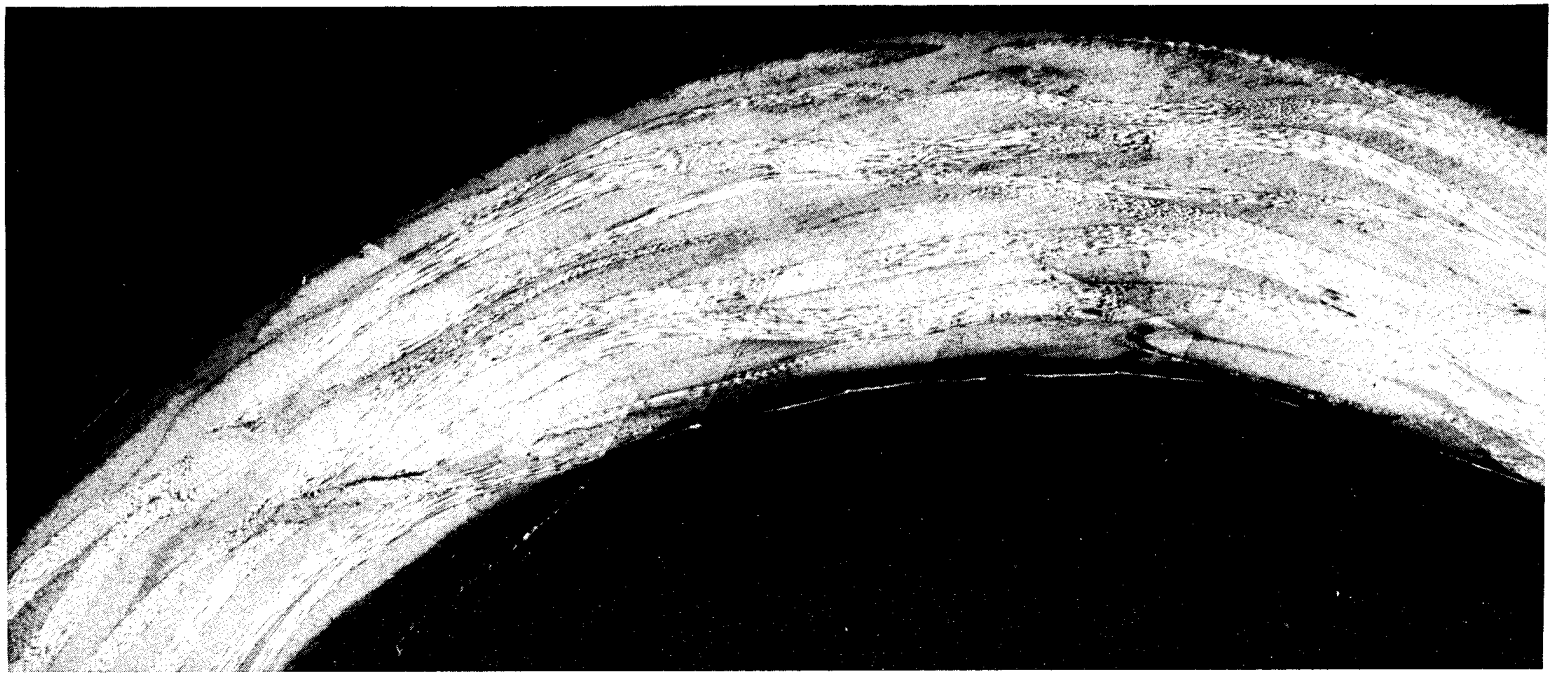


Figure 9

Photomicrograph of Formed 90° Angle Using NTS-20 Trial 1

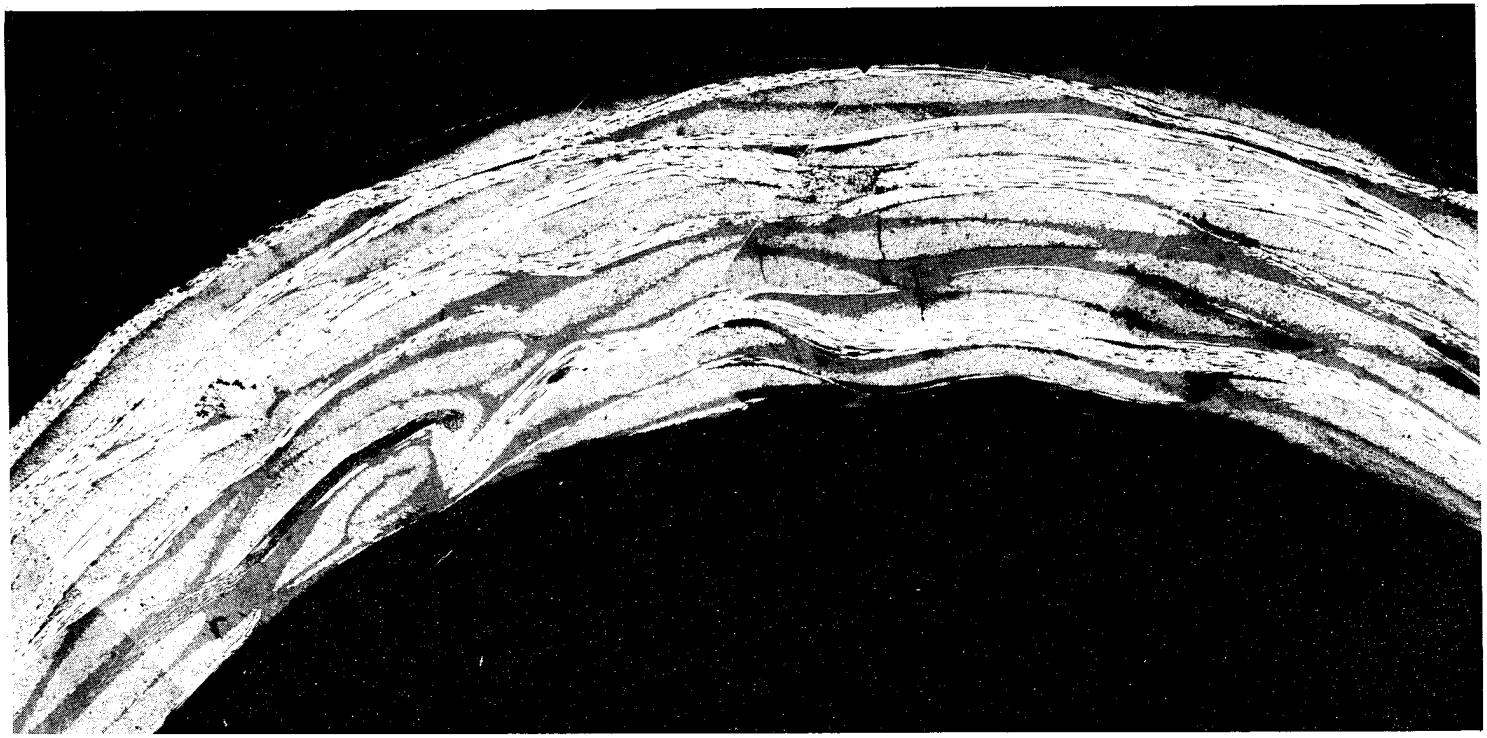


Figure 10

Photomicrograph of Formed 90° Angle Using NTS-20 Trial 2

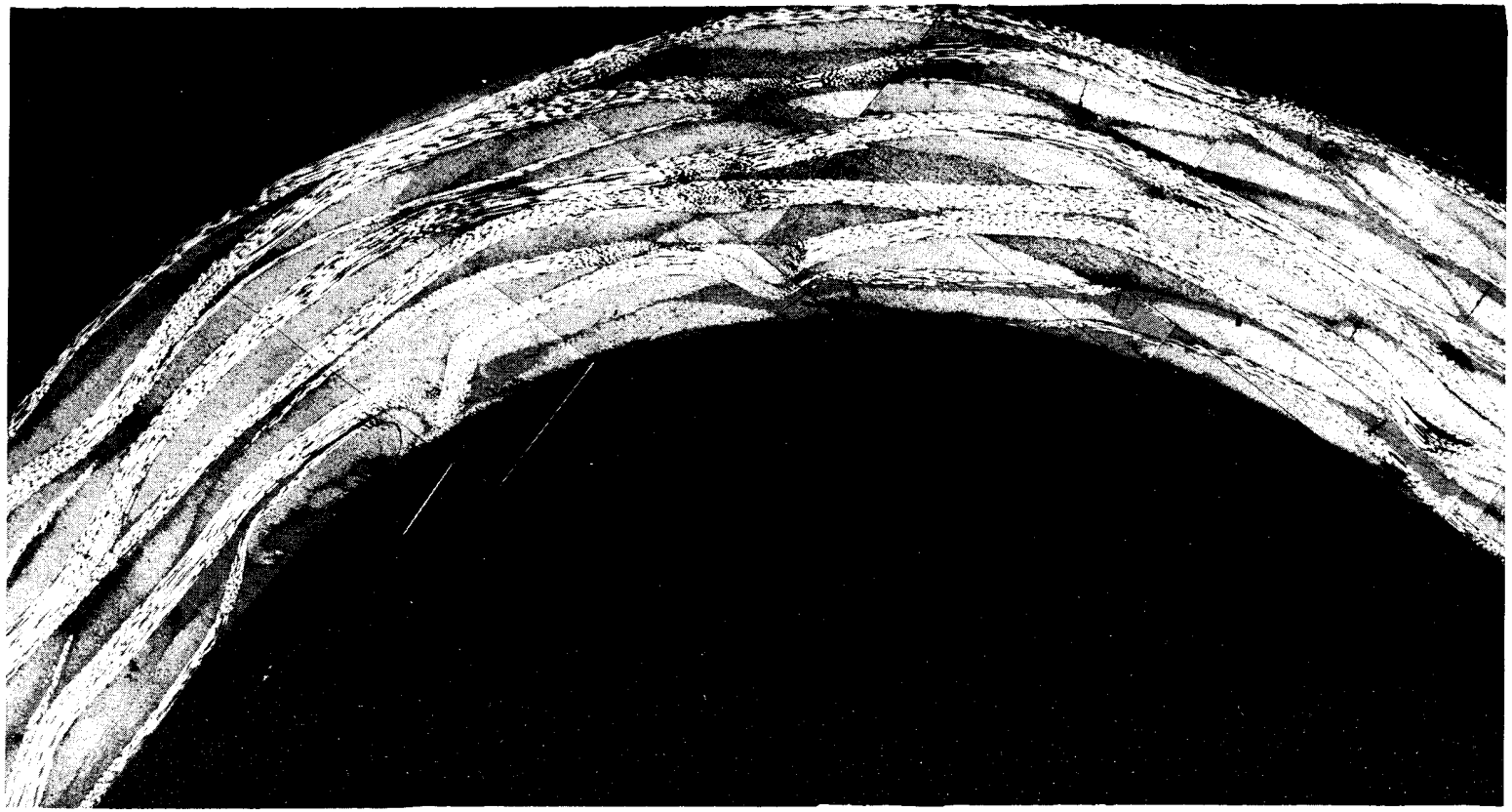


Figure 11

Photomicrograph of Formed 90° Angle Using NTS-20 Trial 3

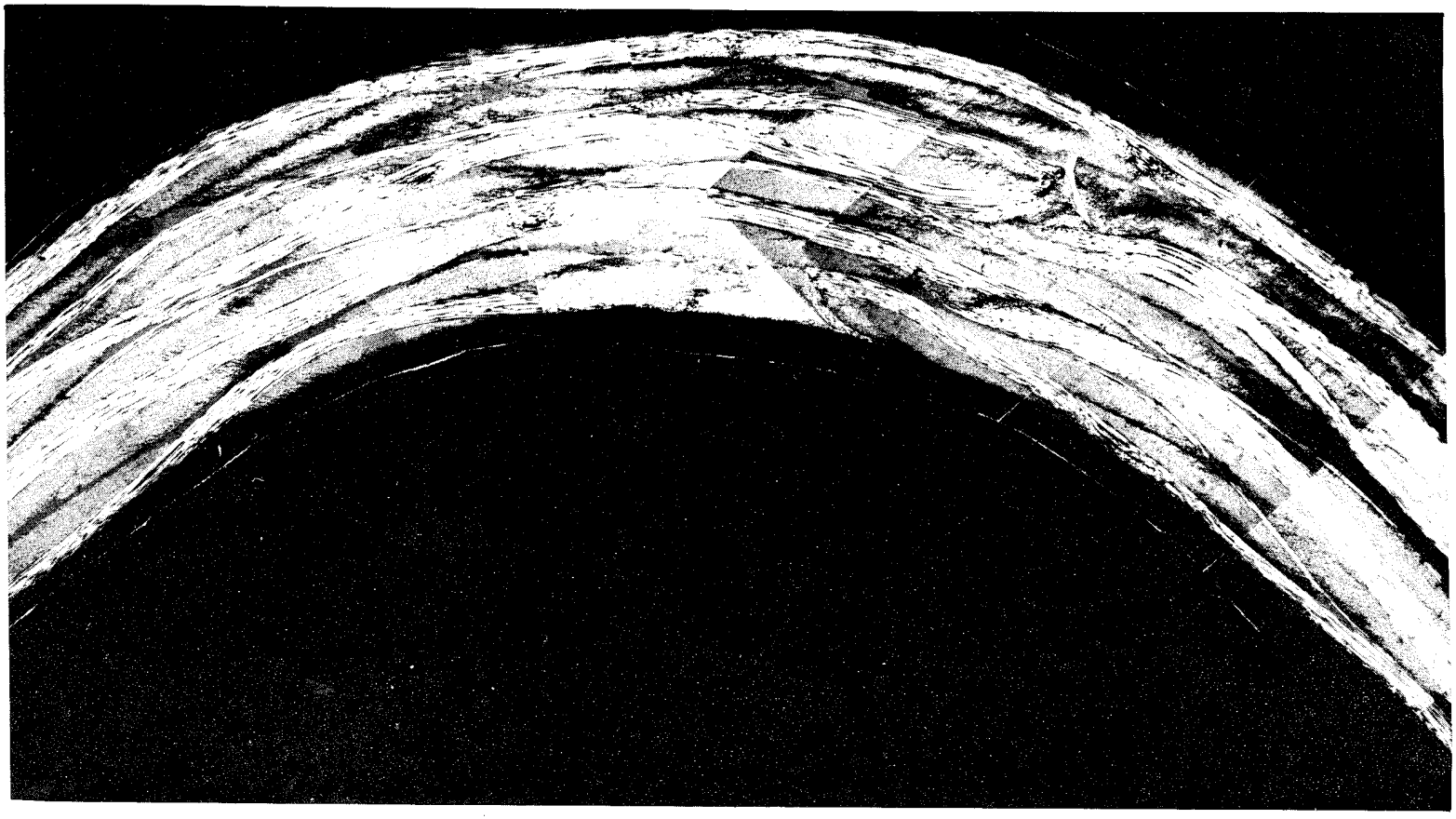


Figure 12

Photomicrograph of Formed 90° Angle Using NTS-20 Trial 4

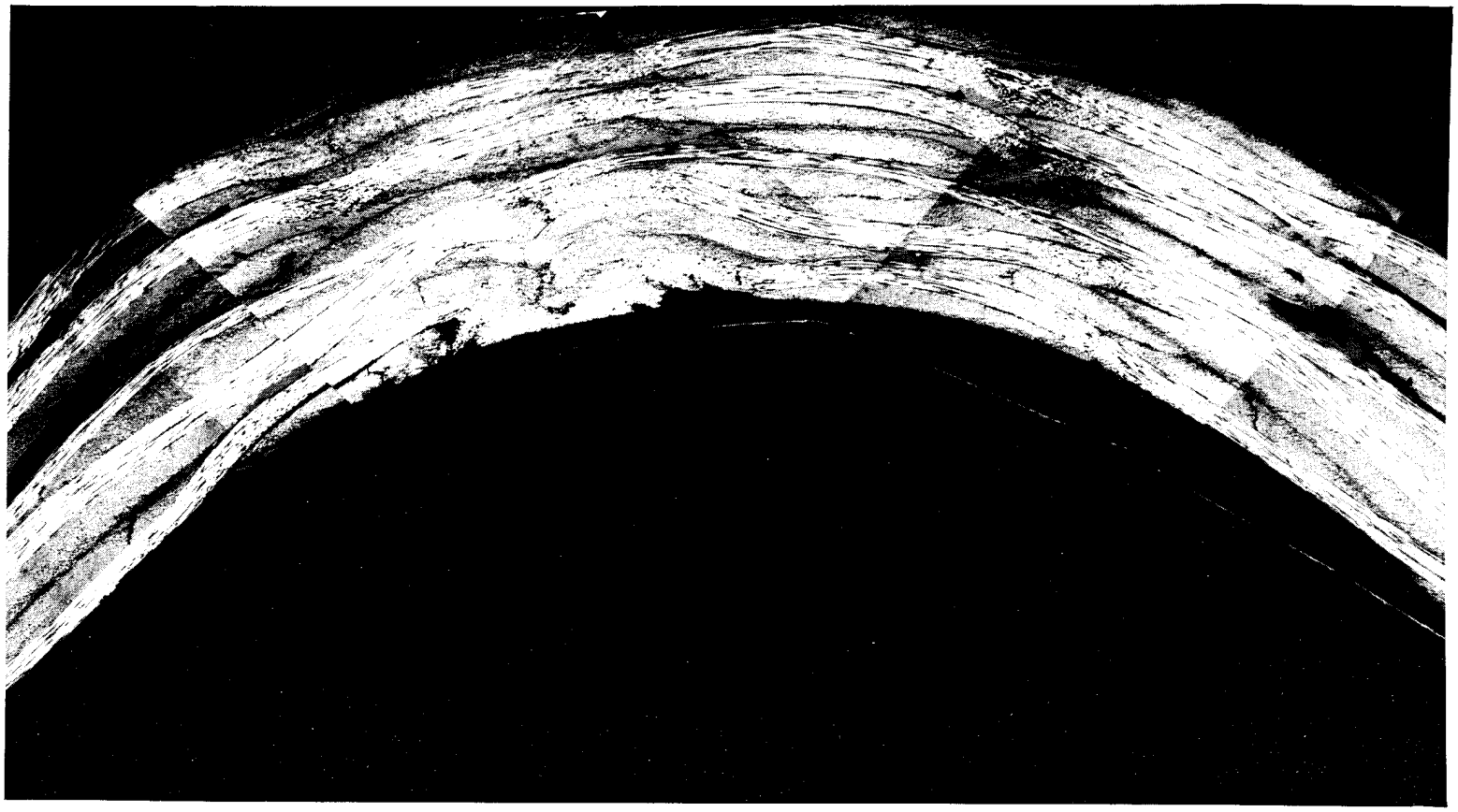


Figure 13

Photomicrograph of Formed 90° Angle Using NTS-20 Trial 5

TABLE VI
SUMMARY POSTFORMING STUDIES

POSTFORMING - MATERIAL

Mold A used with press
Mold B used with vacuum

Panel Code	Starting 3" x 2" Configuration	Postform Mold	Preheat °F	Temp of Mold °F	Pressure Applied or Vacuum	Panel Cure Time	Remarks
PPS	3" x 2" flat panel	flat panel mold A	600	600	VAC	1 hr/600°F	part thickness .075 - .085"
PPS	3" x 2" flat panel	mold B	600	600	200 psi	1 hr/600°F	part thickness .060" (Figure 1)
LaRC	3" x 2" flat panel Trial 1	mold A	750	750	200 psi	2 hr/650°F	part thickness .084 - .090"
LaRC	3" x 2" flat panel Trial 2	mold B	750	750	200 psi	2 hr/650°F	part thickness .060" (Figure 2)
NTS-20	3" x 2" of flat panel	mold A					
	Trial 1		679	724	200 psi	4 hr/650°F	part thickness .060" Figures 3 thru 7
	2		652	710	200 psi	4 hr/650°F	
	3		625	680	200 psi	4 hr/650°F	
	4		658	715	200 psi	6 hr/650°F	
	5		660	675	200 psi	6 hr/650°F	

3.2.2 PPS GRAPHITE COMPOSITE FORMING STUDIES

The processing conditions using PPS had been previously studied on NASC and AFWAL programs. Composites fabricated on this program yielded mechanical properties similar to those previously reported (ref. 1); hence, only forming studies were conducted in this effort. The first forming was done using match die conditions (see Table VI). No resultant fiber damage is evident in the photomicrographs. The second forming was accomplished using vacuum only (see Table VI) with identical results. See Figures 14 and 15 for photomicrographs.

3.2.3 LaRC GRAPHITE COMPOSITE FORMING STUDY

Based on the excellent set of mechanical properties, both control and after stressed aging in solvent sufficient graphite composites were fabricated for forming evaluations. The forming tests were conducted using a 90° angle to match the mold and a platen press. In each case, the mold and composite were heated to a predetermined temperature in the press. The composite was then placed into the mold and the mold closed using 200 psi pressure. The press was then cooled and the part removed from the mold. The formed angle was polished and microscopically examined to determine the effects of the forming operation. Tests using the LaRC 2/graphite were conducted at 650-750°F at 50°F increments. In all trials, fiber breakage did occur along the inner 90° radius. (See Figures 16 and 17 for photomicrographs of the 750°F, 200 psi formed specimen). Based on these data, additional work using LaRC 2 was discontinued because the resin did not meet the program requirements (para. 3.1.1).

3.2.4 PEEK GRAPHITE COMPOSITE STUDIES

In conversation with ICI, it was concluded that due to production limitations and demand for PEEK film, BAC could be furnished with only a limited quantity of PEEK film. With approval of the NASC program monitor it was determined that sufficient data had already been generated by AFWAL and ICI to demonstrate that the PEEK system could be reformed, meeting the program requirements. The forming conditions were 750°F and 100 psi. The supply of film received by BAC was therefore to be used in Task III studies.

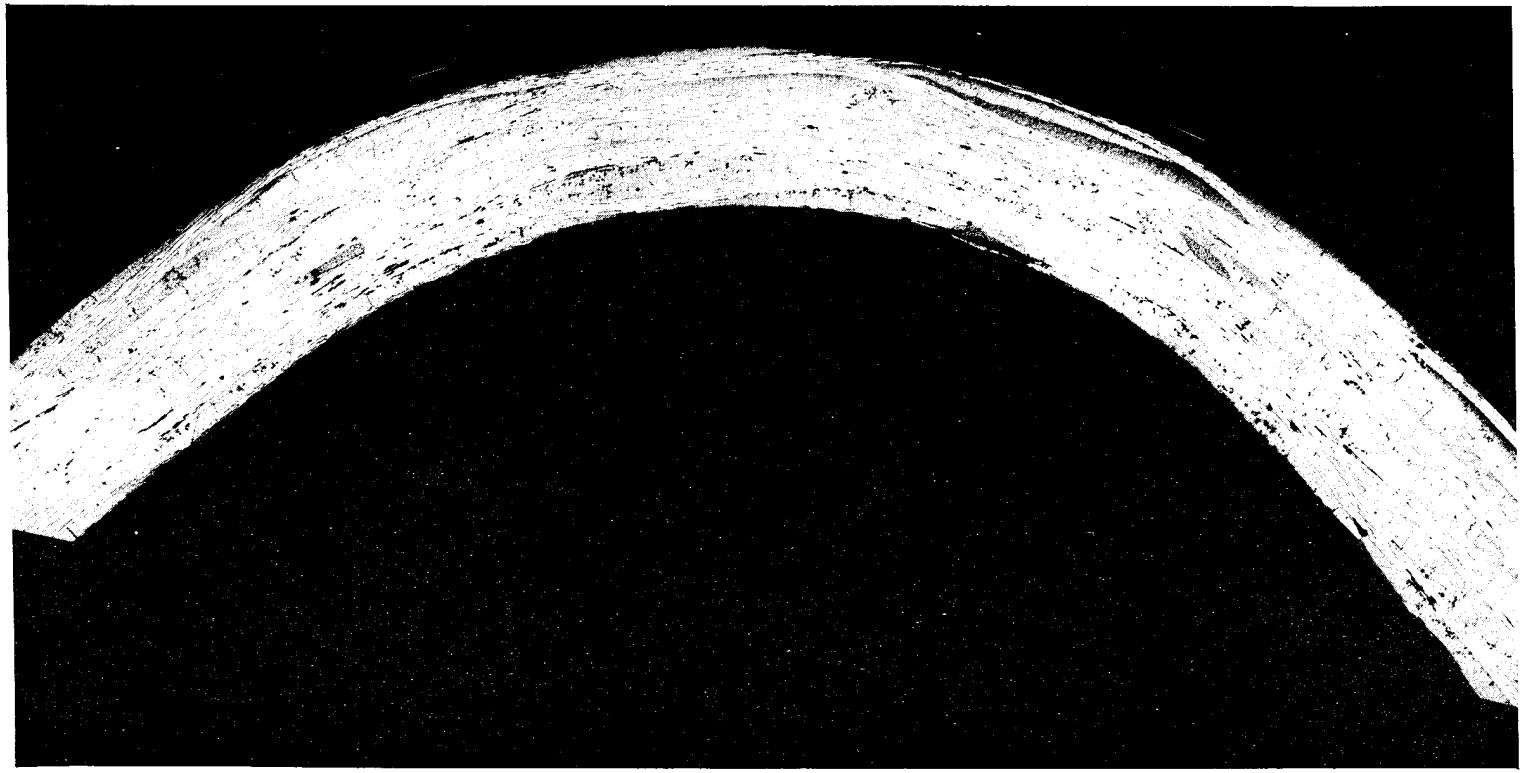


Figure 14

Photomicrograph of Formed 90° Angle Using PPS

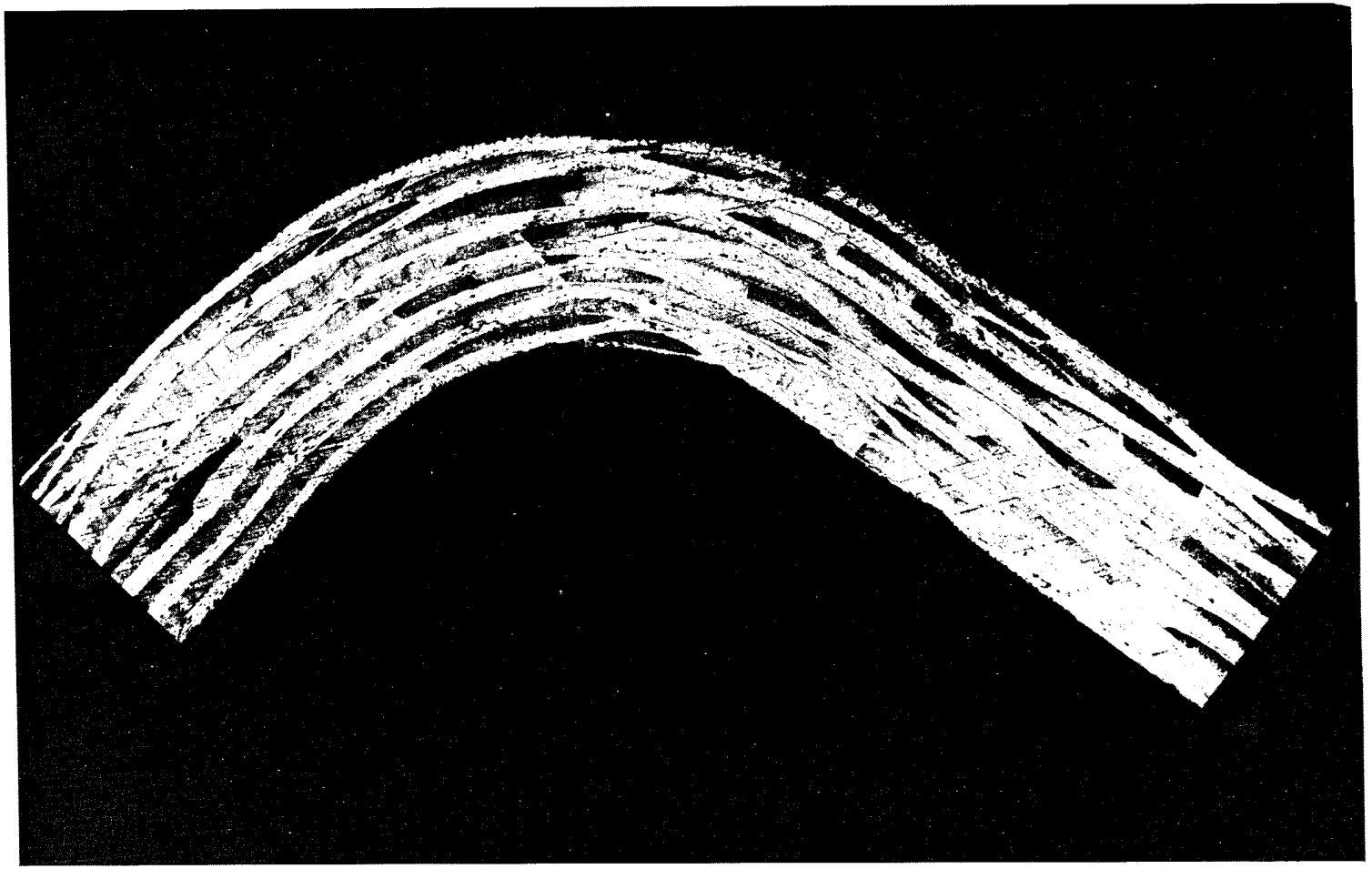


Figure 15

Photomicrograph PPS/Graphite Composite Vacuum Formed 600°F

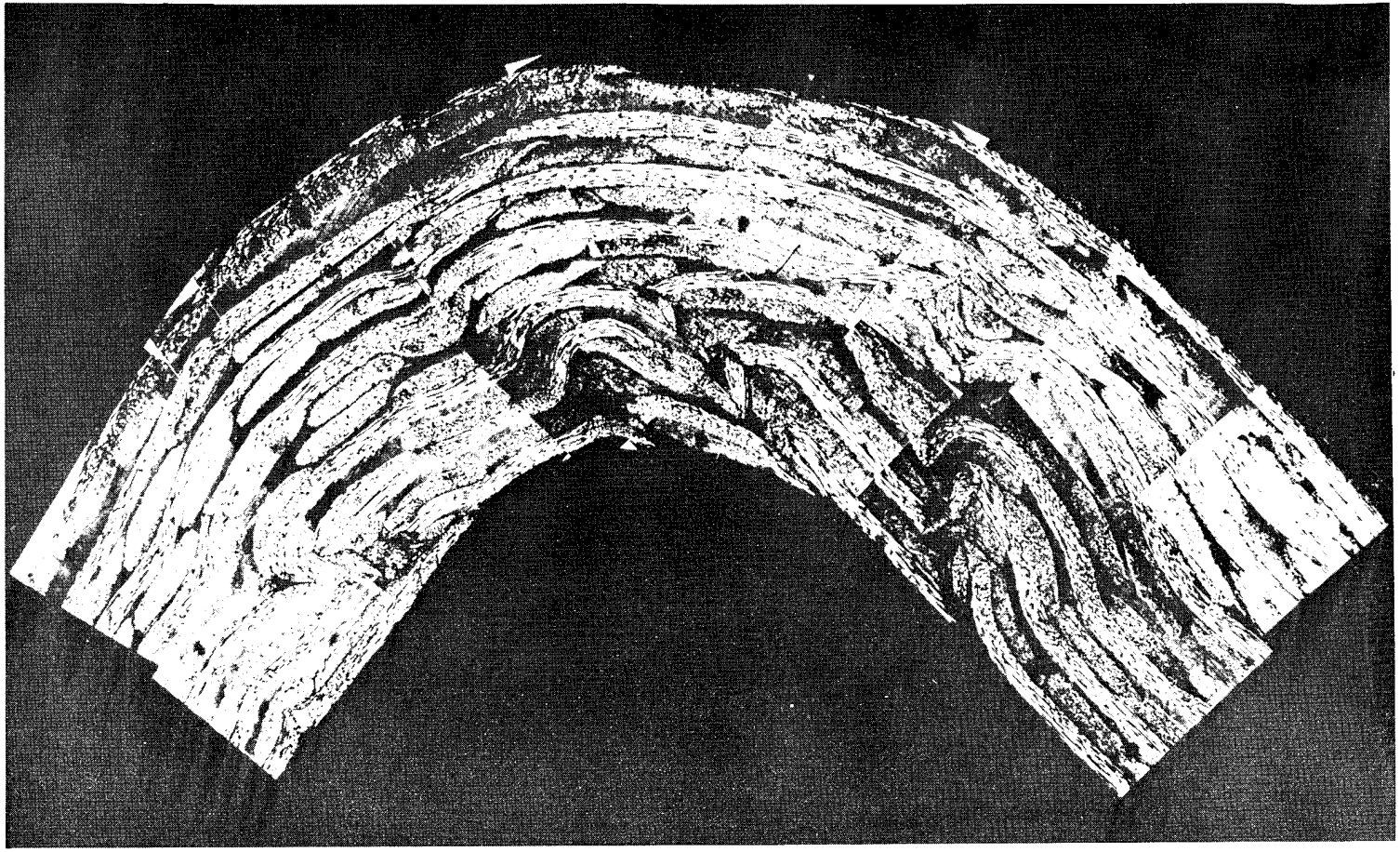


Figure 16
Photomicrograph of LaRC 2/Graphite Composite Formed 750°F, 200 psi

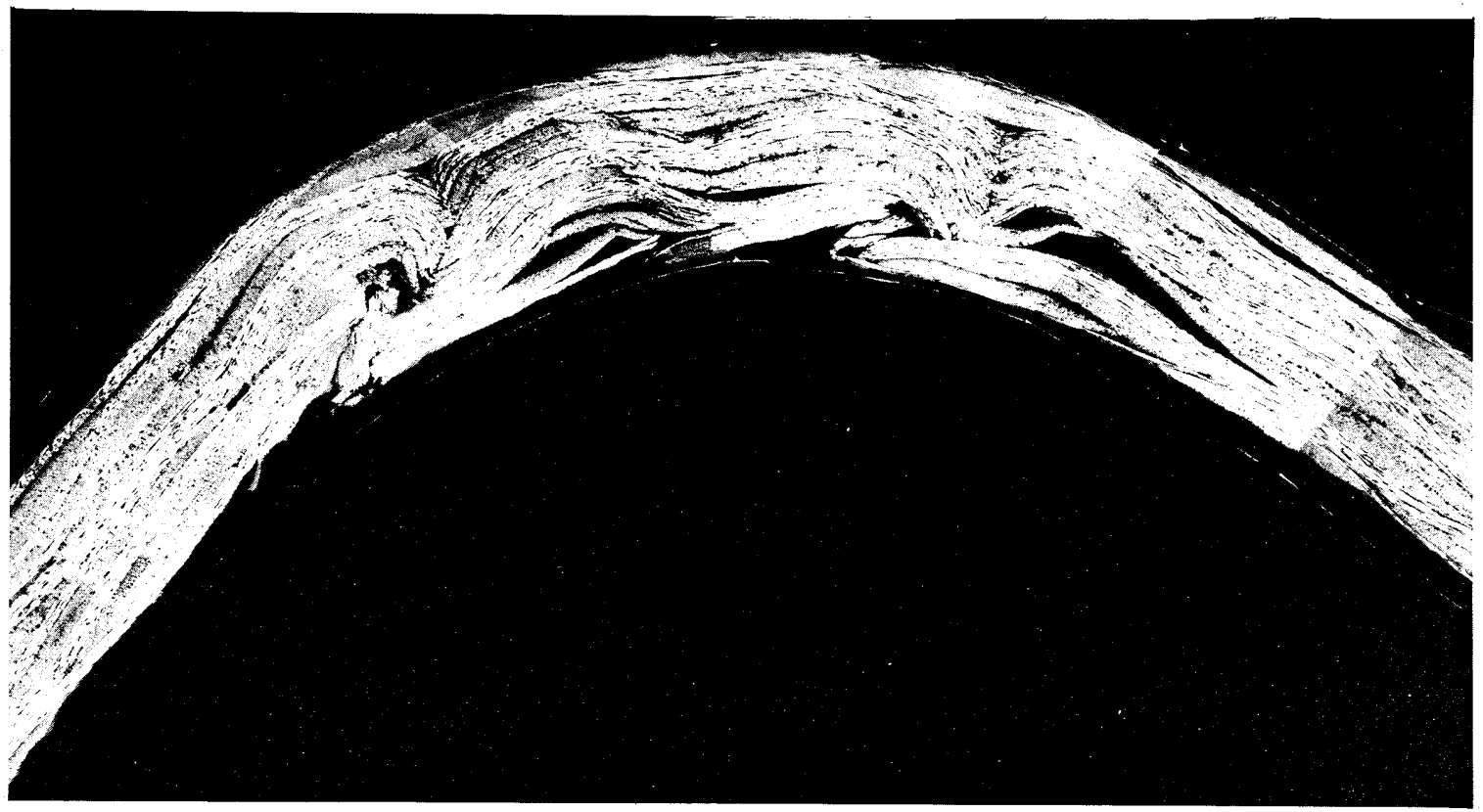


Figure 17
Photomicrograph of Formed 90° Angle Using LaRC 2 Composite

3.3 TASK III—LAMINATE PROPERTIES AND FLUID SCREENING

The objective of Task III was to obtain the mechanical property data necessary to demonstrate that the thermoplastic matrix systems selected from Tasks I and II possessed properties equivalent to the 350°F curing epoxy system, while retaining reformability. A summary of the work accomplished in Tasks I and II is presented in tabular form (Table VII). Three systems were sufficiently promising that the mechanical properties and/or sufficient data from conditioned coupons were obtained to be able to judge the systems performance as compared to conventionally used 350°F cured epoxies (i.e., MY 720 based, eporal cured systems). Some difficulty was encountered in obtaining sufficient data to make the comparison due to availability of PPS and PEEK film. Consequently all tests were not conducted with both of these systems. However it is believed that by careful analysis of the reported data, valid conclusions can be obtained. A more detailed description of the experiments and resulting data is given below.

3.3.1 EVALUATION OF NTS-20 AND PPS IN MAINTENANCE SOLUTIONS

3.3.1.1 Stressed Open Holed Tension/$MeCl_2$

The preliminary evaluation of these systems in paint stripping solutions of $MeCl_2$ and phenol/$MeCl_2$ demonstrated that NTS-20 was susceptible to $MeCl_2$ and phenol based solutions. After lengthy discussions between BAC, Boeing Vertol (user) and the NASC program monitor, it was determined that the immersion of stressed test coupons might exclude an otherwise viable system (NTS-20). Hence it was determined to evaluate the system in another manner, one which more closely represented operating conditions. The test method derived involved the determination of a mechanical property such as tension on a composite specimen of typical layup configuration, i.e., $(0,90,\pm45)_S$, in which a hole had been drilled. $MeCl_2$ was then to be introduced into the hole and maintained for 24 hours. The specimens were then to be tested after allowing sufficient time for the $MeCl_2$ to dry (1 week). To more closely resemble actual operating conditions, the test coupons (open hole tensile specimens) should be stressed at loads approximately 40% of ultimate.

TABLE VII

SUMMARY OF DATA TASK I & II

TEST	PEEK	ULTEM	LARC 2/TPI	NTS-20	PPS	SOTA EPOXY	MODIFIED SOTA EPOXY
Solubility $MeCl_2$	OK	Delam	OK	OK	OK	OK	OK
Stressed Aging $MeCl_2$	OK	—	OK	Marginal	OK	OK	OK
Stressed Aging Phenol/$MeCl_2$	OK	—	OK	Delam	Fair	OK	Good
Forming	N.T.	—	Fiber Breakage 750 200 psi	Fair to good (some fiber damage)	Excellent	N.T. 1/	N.T. 1/
Process	Very Difficult	—	Difficult	Good, cure is long	Excellent	—	
Temp Strength Retention	Excellent	—	Excellent	300°F (Good)	200°F (Good)	220°F	160°F
G_{1C}	—	—	—	3.5-3.7	3.5-4.0	1.8	2.5

1/ Not tested because of the thermosetting nature of the systems and/or supply of resin.

To conduct this test sufficient composite panels were fabricated using NTS-20 and a control, in this case PPS. The open hole coupons were then prepared using both systems and placed in a fixture capable of exerting a load representing 40% of ultimate net tension and $MeCl_2$ introduced into the hole, (see Figure 18). After the conditioning was complete (see Figure 19) the specimens were tested and the results tabulated (Table VIII). The data readily demonstrates that under these conditions both NTS-20 and PPS perform satisfactorily. Using these results and the results from Task I studies, NTS-20 was determined to possess sufficient merit to be evaluated further, even though it possessed some limitations in solvent resistance.

3.3.1.2 Stressed Flexural Tests in Hydraulic Fluids and Fuels

The two systems used in this study were NTS-20 (because of its limited solvent resistance in paint strippers) and PPS. Sufficient graphite composites were fabricated using Appendix A processes to obtain flexural test coupons for these tests. The test coupons were mounted into flexure fixtures and loaded to 40% of the ultimate strength. The fixtures were immersed in the test fluids (i.e., Skydrol, MIL and JP-9 fuel) for 30 days, then tested (Table IX). For comparative purposes, epoxy graphite composite coupons were tested similarly. The thermoplastic systems performed as well as the epoxy systems.

3.3.1.3 Stability to Moisture

In this study the NTS-20 and PEEK system were selected, based on discussion of para. 3.3. The 350°F curing epoxy selected in the study was 3501-5A because of its availability on 3K-70-PW/fabric. The coupon tests selected were matrix dominated (i.e., compression and interlaminar shear). Sufficient graphite composite material was fabricated from each test system, and test coupons were obtained. The specimens were separated and one set tested as cured (control), while the second set was conditioned to saturation at 160°F and 95% relative humidity. (45 days) (see Table X for results). The conditioned thermoplastic systems demonstrated significant improvements over the conditioned epoxy system in elevated temperature tests (i.e., 220°F hot/wet tests).

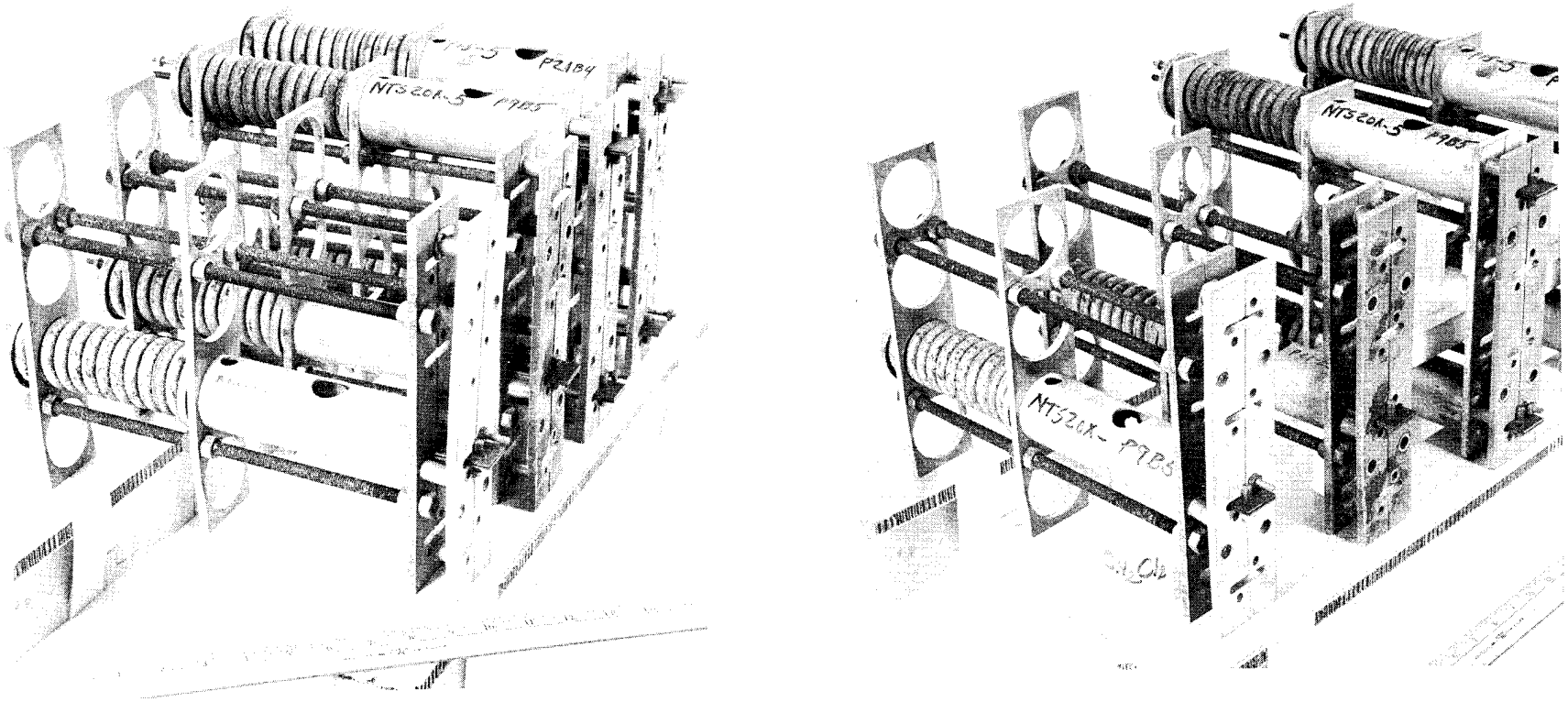


Figure 18

Fixtures for Open Hole Tension Specimens

Methylene Chloride Wicks

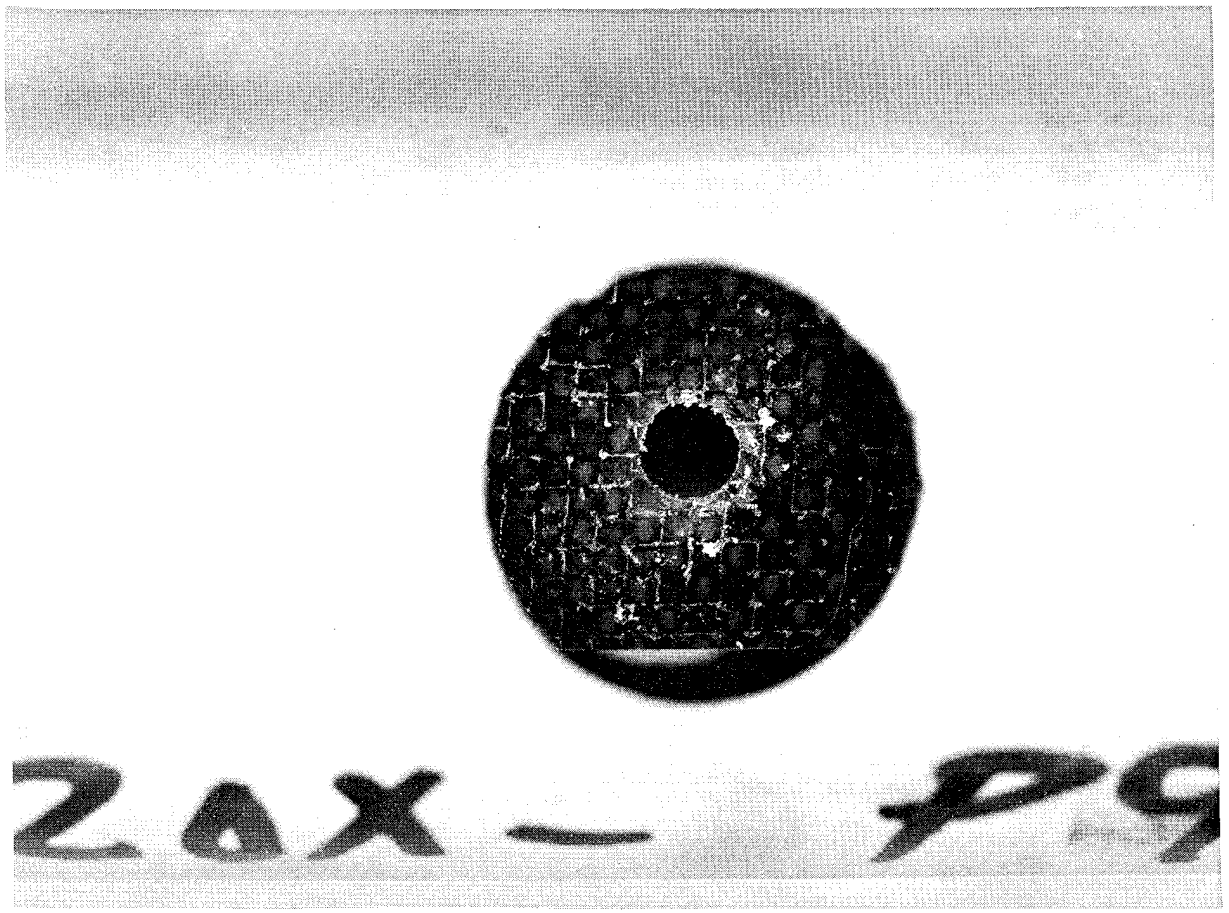


Figure 19
Open Hole Tension
After Solvent Conditioning

TABLE VIII

SUMMARY MECHANICAL PROPERTIES
ENVIRONMENTALLY CONDITIONED THERMOPLASTIC COMPOSITES

Open Hole Tension	(0,90; ± 45)	NTS-20	PPS
Control			
	Ultimate Stress Ksi	35.9	29.9
	Net Stress Ksi	48.2	40.0
1/ Stressed			
	Ult Stress Ksi	40.5	31.9
	Net Stress Ksi	54.4	41.1

1/ Stressed at 40 percent ultimate in saturated $MeCl_2$ for 24 hours. Dried 7 days while under stress, then placed in polyethylene bag until tested.

TABLE IX

SUMMARY MECHANICAL PROPERTIES
FLUID CONDITIONED THERMOPLASTIC COMPOSITES

Flexure	NTS-20	PPS	SOTA EPOXY
Control			
Ultimate Stress/Modulus			
RT	95.1/8.3	67.5/8.5	129.7/9.2
1/ Stressed			
Skydrol Hydraulic Fluid	95.1/8.3	60.4/8.1	123.9/9.7
Mil-H-83282 Fluid	96.6/8.5	66.0/8.3	121.8/9.0
JP-9	119.3/8.6	65.6/8.2	136.2/9.3
Fiber Vol %	60	54	65

1/ Stressed at 40 percent ultimate. Immersed 30 days at ambient conditions.

TABLE X

COMPARISON MECHANICAL PROPERTIES
NTS-20, PEEK, SOTA EPOXY GRAPHITE COMPOSITES

	NTS-20		PEEK		SOTA EPOXY	
Compression Ult/Mod ksi/Msi	Control	Conditioned 1/	Control	Conditioned 1/	Control	Conditioned 1/
RT	57.2/8.0	63.1/8.1	64.5/8.6	62.8/8.5	97.8/9.3	53.1/9.1
220°F	54.5/8.0	49.0/8.2	59.1/8.4	57.2/8.5	60.1/8.9	28.6/8.2
300°F	36.4/7.4	27.0/6.9	45.4/5.9	46.3/6.1	N.T. 2/	N.T. 2/
Short Beam Shear						
RT	8.6	8.5	7.5	7.9	10.0	6.2
220°F	6.0	5.8	6.8	6.7	5.8	2.4
Weight Gain %	0.9		0.7		1.9	
Fiber Volume	55		60		64	

1/ Humidity conditioned 45 days at 160°F and 95% RH

2/ Coupons not tested

Fabric used: Epoxy sized 3K-70-PW

3.3.2 PRELIMINARY DESIGN PROPERTIES NTS-20, PPS, AND PEEK GRAPHITE COMPOSITES

The overall objective of Task III studies was to obtain sufficient design data to determine the utility of thermoplastic systems in airframe structure. Toward this end sufficient graphite composites were fabricated to accomplish the testing given in Table XI for PPS and NTS-20 systems. The PPS composites were made using 2 mil PPS film furnished by the Phillips Petroleum Company and 3K-70-PW (epoxy sized) graphite fabric. The composites were consolidated using autoclave molding procedures described in the third quarterly technical report. The NTS-20 composites were fabricated using resin made by Raylo prepregged by U.S. Polymeric on 3K-70-PW graphite fabric. Due to the quality of some of the graphite prepreg received from U.S. Polymeric, the material was reprocessed at Boeing prior to consolidation using autoclave molding procedures reported. Because of the limited supply of PEEK film only some of the PEEK test coupons could be fabricated. The PEEK composites were made similarly to the PPS in that the 2 mil PEEK film was interleaved between the dry graphite fabric (i.e., 3K-70-PW T300) and consolidated using press molding procedures described in the Appendix A. Test results are given in Table XII.

3.4 TASK IV—FIBER/RESIN INTERFACE STUDY

During this task, the interfacial attachment of NTS-20 to Kevlar reinforcement was studied. The studies were conducted to aid in the selection of a Kevlar sizing to be used on the Boeing Vertol manufacturing technology program. The specific fiber reinforcement selected for evaluation was 353 style, crow foot weave. The surface treatments selected included:

No Finish
HR 600 Polyimide Coated
Skybond 703 Polyimide Coated

TABLE XI

TASK III TEST MATRIX FOR
DESIGN PROPERTIES OF SELECTED RESINS

Property		Test Temperature RT	300°F
Tension strength and modulus			
$(0,90;\pm 45)_S$	8 ply	5	5
$(\pm 45)_2$	8 ply	5	5
Compressive strength and modulus			
$(0,90;\pm 45)_S$	12 ply	5	5
Fatigue $(0,90;\pm 45)_S$ 1/		5	5
Impact Resistance		5	-
G_1C		5	-
Resin content, specific gravity Void content, fiber volume		3	

1/ - Specimens fatigued 500,000 cycles minimum in compression, then tested to ultimate failure determining ultimate stress, modulus and strain properties.

TABLE XII

DESIGN PROPERTIES OF SELECTED RESINS

Property	Laminate Configuration		Thermoplastic System					
			PPS		NTS-20		PEEK 1/	
			RT	200F 2/	RT	300F	RT	300F
Tension								
Ultimate stress/Modulus (Ksi/Msi)	$(0,90,\pm 45)_S$		37.1/5.63		65.0/6.43	58.8/5.7	70.0/7.1	62.3/7.0
Ultimate stress/Modulus (Ksi/Msi)	$(\pm 45)_{2S}$		18.1/1.88	15.8/1.10	29.9/2.45	19.1/1.28	1/	1/
Compression								
Ultimate stress/Modulus (Ksi/Msi)	$(0,90;\pm 45)_S$		34.0/5.9		56.4/5.8	48.2/5.9	62.1/6.0	52.3/6.1
Fatigue Ultimate Stress Ksi initial/residual	$(0,90;\pm 45)_S$		42.3/38.0		3/		1/	1/
Impact Resistance	$(0,90;\pm 45)_S$	P_f lbs Force 4/	845		954		1/	1/
G_1C	$(0,90)_S$	inch lbs	3.7	—	3.5	—	4.0	—
Resin Content			35.2		33.0		34.0	

1/ Data not obtained due to lack of resin film

2/ PPS system capability limited to 200F

3/ Testing apparatus not capable of 300°F testing

4/ Defined as the peak load where fiber failure and/or resin failure occurs. In some cases a potential matrix failure could have occurred at lower values.

As indicated two surface treatments were evaluated, the number and treatments selected to complete the study initiated under previous NASC programs.

Prepreg was made using 10% solids NTS-20 resin from the Raylo batch of material. The two polyimide sizings were applied to Kevlar fabric which was previously dried 1 hour at 350°F. The polyimide sizings were applied to the dried Kevlar fabric at approximately 1.5% resin content with solutions of HR 600 and Skybond 703. The sizing solution was coated onto the fabric and the impregnated fabric was vacuum bagged to insure wetting of the Kevlar fibers. The prepreg was then dried in an air circulating oven until the polyimide resins were cured. The NTS-20 resin varnish was coated onto the sized fabric and dried. A composite laminate was then fabricated using procedures described in Appendix A. The composites were then machined into flexure and interlaminar shear specimens and properties were determined. The data (Table XIII) was compared to the data previously generated in ref. 1 program. Representative failed flexure specimens were studied using photomicrographs; these indicated that the sizing systems performed as well as previously studied sizings.

3.5 TASK V—PRELIMINARY DESIGN DATA USING NTS-20 THERMOPLASTIC RESIN

The objective of this task was to develop some preliminary design data using NTS-20 resin manufactured by Raylo and prepregged onto broad goods using 3K-70-CS graphite fabric and style 343 crow foot satin (epoxy sized) Kevlar fabric. Some difficulty was encountered during this effort but sufficient data was obtained to demonstrate that the NTS-20 system could be used successfully in the work platform structure. Sufficient resin was manufactured by Raylo Chemical Company to supply prepreg broad goods to the referenced program. Vertol selected Fiberite to prepreg the Raylo produced resin onto graphite and Kevlar reinforcements for the manufacturing technology program. BAC was given the responsibility of performing quality control tests on the NTS-20 resin and the NTS-20 broad goods received from Fiberite. Using the quality control data, a consolidation process was generated to produce the sheet stock to be used at Vertol. During the course of the program significant technical and/or non-technical probelms were encountered that caused delays in both this program and Vertol's program (ref. 4). They could be summarized as:

1. Availability of raw materials at Raylo for NTS-20 resin.
2. Reproducibility of NTS-20 resin.
3. Broadgoods received from Fiberite.

TABLE XIII

SUMMARY MECHANICAL PROPERTIES
FIBER SIZING STUDY 3/

Matrix	Reinforcement	Fiber Finish	Mechanical Properties Flexural Strength ksi		Flexural Modulus Msi		ILS ksi	
			RT	300°F	RT	300°F	RT	300°F
NTS-20	Kevlar Fabric	None	47.6	38.7	5.2	5.2	5.2	3.8
		Epoxy Compatible 1/	35.6	33.1 2/	3.9	4.5	4.8	4.1
		Plasma Sprayed 1/	39.6	28.7 2/	4.1	4.1	3.0	3.0
		PMR-15 1/	37.7	30.9 2/	4.6	4.3	3.8	3.0
		HR600	45.5	39.5	4.6	5.1	4.7	3.7
		Skybond 703	44.9	37.2	4.4	4.3	5.2	3.7

1/ Data extracted from NASC Contract (N00019-080-C-0365)

2/ On previous program elevated temperature data obtained at 250°F

A more detailed discussion of those problems and of the data generated during this task are given below.

3.5.1 Non Technical Problems

Materials procurement for the NTS-20 broadgoods to be used in this task was the responsibility of Vertol under their manufacturing technology program. Toward this end a competitive procurement was awarded to Fiberite for the program prepregs. Fiberite determined that they could not manufacture the NTS-20 resin and in turn contracted with Raylo Chemical Company for three hundred pounds of resin. When Raylo attempted to procure the raw materials (i.e., dichlorodiphenylsulfone and bisphenol A) they found the dichlorodiphenyl sulfone unavailable in the United States. A supplier was located in Europe and sufficient dichlorodiphenyl sulfone was procured for the program.

This incurred approximately a six month delay in both programs. However, once the material became available Raylo, in conjunction with BAC, developed and tested manufacturing procedures for 100 pound batches of NTS-20 resin (see Table XIV for chemical data). Samples of the Fiberite NTS-20 resin and NTS-20 resin for the remaining portion of this program were supplied to BAC Quality Control for chemical characterization (see Table XV). Finding the material to be essentially the same as laboratory made NTS-20, three hundred pounds of resin was supplied to Fiberite and one hundred pounds to BAC (lots 1080 A3, A4, A5 and 1080 A2 respectively).

3.5.2 QUALITY CONTROL OF NTS-20 RESINS (CHEMISTRY)

The Quality Control of the Raylo NTS-20 resin provided to Fiberite (i.e., lots 1080 A3, A4 and A5) was accomplished in the BAC Quality Control laboratory. The results are given in Table XVI. Based on these data the material appears to be similar to the BAC standard and the 100-pound lot (i.e., lot 1080 A2) of resin previously received by Boeing.

TABLE XIV

SUMMARY OF DATA NTS20
RESIN FROM RAYLO

GPC Analysis	Avg Mol WT	No. Avg Mol WT	Polydispersitive Factor
Raylo Batch	21,169	4,705	4.5
BAC Standard	19,920	4,480	4.1

IR Analysis	Imide 1/ Aromatic-0-Aromatic	Carbonyl Aromatic-0-Aromatic	Sulfone Aromatic-0-Aromatic
Raylo 100 lb Batch	15.2	7.5	73.9
BAC Standard	11.8	4.5	82.1

1/ Ratio of wavenumbers characteristic of absorption band

TABLE XV

SUMMARY CHEMICAL DATA
NTS-20 RESIN SUPPLIED BY RAYLO
GPC ANALYSIS

	Wt Average Mol Wt	No. Average Mol Wt	Poly dispersitive Factor
BAC Standard	19,920	4480	4.1
1080A2	21,169	4705	4.5
1080A3	21,580	3955	5.5
1080A4	24,373	5269	4.6
1080A5	24,222	4811	5.0

IR ANALYSIS

	Imide 1/ Aromatic-O-Aromatic	Methyl Aromatic-O-Aromatic	Sulfone Aromatic-O-Aromatic
BAC Standard	11.8	—	82.1
1080A2	15.2	—	73.9
1080A3	7.7	16.5	75.0
1080A4	9.4	16.6	74.4
1080A5	7.0	15.2	76.2

1/ Ratio of wavenumbers characteristic of absorption band

TABLE XVI

QUALITY CONTROL DATA
GRAPHITE COMPOSITE MADE FROM RAYLO NTS-20

	BAC Standard	1080 A2	1080 A3		1080 A4		1080 A5	
Flexural Ultimate Ksi				2/		2/		2/
RT	93.0	95.2	76.1	67.9	86.0	89.8	40.0	26.7
300°F	56.3	70.0	18.7	—	25.3	—	13.9	—
RT 1/	—	—	39.2	—	39.9	—	20.8	—
Flexural Modulus Msi								
RT	7.2	7.7	7.06	9.04	8.89	8.77	7.60	7.73
300°F	7.4	7.9	4.82	—	6.45	—	3.91	—
RT 1/	—	—	6.41	—	5.95	—	5.11	—
Interlaminar Shear Ult. Ksi								
RT	6.5	9.5	5.58	6.24	6.02	7.49	5.10	3.54
300°F	5.1	5.6	3.46	—	2.54	—	1.47	—
RT 1/	—		2.91	—	—	—	2.40	—
Resin Content %	33.0	35.0						
Specific Gravity g/cc	1.55	1.53						
Fiber Volume %	59	56.5						
Void Volume %	<1	<1						

1/ Immersed in $MeCl_2$ for 90 hours, then air dried 2 hours prior to testing.

2/ Additional 3 hour cure at 650°F.

3.5.3 QUALITY CONTROL OF NTS-20 RESIN (COMPOSITES)

Graphite prepreg was made using the resins supplied by Raylo and epoxy sized 3K-70-PW T300 graphite cloth. From this prepreg, composite panels were fabricated, machined and tested. The results are summarized in Table XVI. Based on these data, lots 1080A3 and 1080A4 appeared comparable to the previous Boeing lot (1080A2) and the BAC standard. However, lot 1080A5 differed significantly from any previous lots. On the chance that the "A5" lot had not been cured sufficiently, additional panels were postcured three hours further at 650°F. The test results are also reported in Table XVI. The additional cure time didn't appear to affect the results, which still indicated that lot 1080A5 was different.

3.5.4 QUALITY CONTROL OF FIBERITE'S MATERIAL

Based on the initial screening of the three lots of NTS-20 resin, Fiberite prepregged 10 yards of style 343 graphite fabric with lot 1080A3, and provided 5 yards to BAC for Quality Control testing. Because of funding limitations, only the composite properties of this material were obtained (Table XVII). Two sets of panels were made, one set cured with a bleeder material in contact with the prepreg and the other without a bleeder. The panel made using a bleeder material was then cut in half and one portion postcured an additional 3 hours at 650°F. The data presented in Table XVII indicates that the additional 3 hours at 650°F improved the properties.

Based on this data, Fiberite then provided BAC with 20 yards of style 343 graphite/NTS-20 and style 181 Kevlar/NTS-20 prepreg made using resin lot 1080A3. Quality Control panels were fabricated, again using a no bleed configuration and a bleed configuration. In this case it appeared that the extra time at 650°F improved the properties, but not to the same extent as for the pilot plant prepreg (Table XVIII). The data obtained using the Kevlar was comparable to the data obtained using the same fabric style on Contract N00019-80-C-0365 (i.e., 4.0 ksi vs. 4.8 ksi for short beam shear test coupon).

TABLE XVII

QUALITY CONTROL OF PREPREG FROM FIBERITE 1/

	Bleed		No Bleed
	6 Hours	9 Hours	
Flexural Ultimate Ksi			
RT	22.51	67.39	22.94
300°F	6.08	17.71	9.30
Flexural Modulus Ksi			
RT	6.84	7.85	6.63
300°F		4.87	6.17
Inerlaminar Shear			
RT	1.60	3.90	1.93
300°F	N.T.	1.68	N.T.
Resin Content	29.3		28.6
Fiber Volume	61.6		62.0
Specific Gravity	1.52		1.52
Void Volume	4.1		4.5

1/ 5 Harness Satin epoxy sized T300

NOTE: Typical epoxy composites using same fabric give:
RT Flexural Stress 90,000 to 100,000 Psi and,
ILS of 6.5-7.0 Ksi.

TABLE XVIII

QUALITY CONTROL OF PREPREG FROM FIBERITE 1/

	Graphite				Kevlar
	No Bleed		Bleed		Bleed
	6 Hour	9 Hr	6 Hour	9 Hr	9 Hr
Flexural Ultimate Ksi					
RT	22.9	35.2	22.5	48.0	33.4
300°F	9.3	18.9	—	15.7	29.4
Flexural Modulus Msi					
RT	6.63	5.83	6.84	8.27	4.69
300°F	6.17	1.80	—	5.44	4.80
Interlaminar Shear Ksi					
RT	1.9	3.0	1.6	3.0	4.0
300°F	0.7	0.8	0.4	2.1	3.9

1/ Style 343, 3K-70-CSW epoxy sized T300 - 20 yds
Style 181 Kevlar

3.5.5 TECHNICAL PROBLEMS

Based on data generated in the above tasks, additional chemistry tests were conducted on the prepregs made by Fiberite (see Table XIX). Based on these data it was determined that the resin extracted from the Fiberite prepregs was different from the standards of like resin received at Boeing. It was concluded that the resin was significantly changed by the Fiberite process and upon lengthy discussions with Fiberite it was concluded that the solvent system used (i.e., NMP) was the most probable culprit. Additional prepregs prepared at Fiberite using only tetrahydrofuran (THF) solvent were received by BAC and evaluated (Table XX). The results of these tests indicated that the original chemical properties of the resin were not altered. The materials were then used to obtain selected design properties.

3.5.6 SELECTED DESIGN PROPERTIES

The significant problems encountered with the prepregs obtained from Fiberite dictated that only selected design properties could be obtained before this program ended.

The laminate configuration selected was the all Kevlar (seven plies) and graphite/Kevlar hybrid (face plies of Kevlar and core of six plies of graphite) all at (0,90) orientation.

The tests selected were compression, interlaminar shear and rail shear with one set of specimens as control and a second set of specimens from laminates conditioned unsupported in an aircirculating oven for 2 minutes at 550°F. These conditions simulate the processing temperatures and times for Vertol's vacuum forming operation.

The coupons were tested and the results are tabulated in Table XXI.

TABLE XIX

MOLECULAR WEIGHT DETERMINATIONS

NTS-20

	Powder	Soln	Prepreg	1080-A-3	1080-A-4	1080-A-5
	4	5	6	A 1	B 2	C 3
Avg Mol WT	21.8	21.1	22.85	19.90	24.67	21.76
No. Avg Mol WT	2.28	2.07	2.28	2.22	2.79	2.84
Polydispersive	8.5	10.2	10.0	8.95	8.84	7.66
Z Average	219	220.4	246.2	188.3	235.7	207.8
	Union Carbide Sample			BAC		
	C 72.85	73.64	73.80	C 66.9	66.4	64.3
	H 5.12	5.21	5.7	H 5.44	5.40	5.28
	N .52	.51	.52	N .128	.127	.129
	35					
	C 72.62	72.55	72.76	C 64.2	67.3	68.2
	H 5.10	5.07	5.27	H 5.0	5.21	5.39
	N .43	.41	.45	N .209	.229	.204

TABLE XX

NMP, THF PREPREGGED NTS-20 COMPOSITE DATA

	Kevlar		Graphite	
Mechanical Property	NMP	THF	NMP	THF
Flexure				
Ultimate ksi				
RT	33.4	38.3	48.0	54.4
300°F	29.4	27.2	15.7	25.8
Modulus Msi				
RT	4.7	3.6	8.3	7.1
300°F	4.8	3.8	5.4	7.5
Short Beam Shear				
Ultimate ksi				
RT	4.0	4.7	3.0	3.6
300°F	3.9	2.7	2.1	1.1

TABLE XXI

SELECTED DESIGN DATA

Property	Hybrid Laminates $(0^o\ K/0^o\ Gr\ 3)_S$ 1/		Kevlar Laminate $(0^o\ K_7)$	
	Control	Conditioned	Control	Conditioned
Tension				
Ultimate ksi	67.2	62.3	44.0	43.4
Modulus Msi	6.7	6.5	4.6	4.7
Compression				
Ultimate ksi	61.7	58.9	39.5	39.1
Modulus Msi	5.8	5.8	4.0	3.8
Short Beam Shear				
Ultimate ksi	5.42	5.26	4.76	4.50
Rail Shear				
Ultimate ksi	15.2	15.3	12.3	12.0
Modulus Msi	0.47	0.43	0.32	0.34

1/ Kevlar Style 343 Crowfoot Satin; Graphite 3K-135-8H T300 Fabric

4.0 CONCLUSIONS AND RECOMMENDATIONS

The primary objective of this program was to develop graphite reinforced thermoplastic composites with fluid resistance and mechanical properties equivalent to 350°F curing epoxy resins. The studies screened five commercially and/or near commercially available thermoplastics, four of which successfully passed the fluid screening tests. The LaRC-2 system, however, failed to meet the established thermoforming criteria, leaving three resins which met the general program requirements. Resin availabilities dictated that only the NTS-20 could be completely characterized.

The secondary objective of improving interfacial attachment between polymer and reinforcement was successfully accomplished for the NTS-20 system on Kevlar fabric using HR600 or Skybond 703 as surface finishes. The other secondary objective to obtain point design numbers using NTS-20/Kevlar and Kevlar-graphite hybrid composites from commercially produced NTS-20 resin and prepreg, yielded properties essentially equivalent to those of SOTA epoxy systems. More detailed conclusions are presented in Section 4.1, and the recommendations for further study/applications are presented in Section 4.2.

4.1 CONCLUSIONS

- Five thermoplastic resins were evaluated in graphite and/or Kevlar composites.
- PEEK, PPS and NTS-20 resins were selected for additional characterization.
- Design oriented properties of candidate thermoplastics were equivalent to SOTA epoxies; PEEK gave the best properties, then NTS-20 and PPS.
- The ease of manufacture of the candidate systems in descending order was PPS, NTS-20 and PEEK.
- Environmental stability of all the thermoplastics were equivalent to SOTA epoxies; PEEK, PPS and NTS-20 possessed the best overall properties.
- The point design numbers of NTS-20/Kevlar and NTS-20/Kevlar-graphite hybrid composites were equivalent to those of SOTA epoxies, and sufficient for CH-46 work platform applications.
- NTS-20 resin and NTS-20 broadgoods were obtained from commercial sources.
- PEEK, NTS-20 and PPS systems appear suitable for use in graphite reinforced composite structure for Naval aircraft.

4.2 RECOMMENDATIONS

Based on the results of evaluations performed during this program, the following recommendations are offered:

- o Continue evaluation of newly developed thermoplastic polymers as matrix materials for graphite and Kevlar reinforced composites.
- o Develop manufacturing procedures that would demonstrate fast forming on simulated aircraft structural components. Prime candidate structures for these procedures would be skin stiffener stringer panels used in aircraft fuselage structure.
- o Demonstrate the structural property superiority of these skin stringer panels to SOTA epoxy-graphite structure.
- o Demonstrate that the post buckling fatigue behavior of the skin stringer panels is equivalent or superior to that of SOTA epoxy composites.
- o Evaluate the structural properties of NTS-20/Kevlar-graphite honeycomb structure.
- o Obtain more detailed design properties with PEEK, NTS-20 and PPS resin systems.

REFERENCES

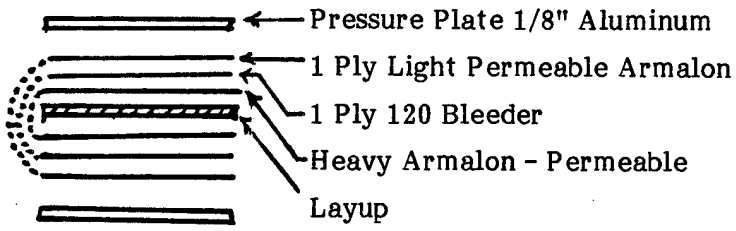
1. Contract N00019-79-C-0203, "Graphite Reinforced Thermoplastic Composites."
2. Contract N00019-82-C-0233, "Evaluation of Thermoplastic Composites and Fabrication Techniques."
3. Contract N00019-82-C-0383, "Development of Improved Modified Polysulfone Resins."
4. Vertol's Manufacturing Technology Program, NASC Contract N00019-79-G-0332, "Demonstrate Representative Tooling and Fabrication Concepts for Load-Cost Reinforced - Plastic Design for CH-46 Hinged Work Platform."
5. Boeing Unpublished Data.

APPENDIX A

General Processing Procedures for Graphite Reinforced Composites

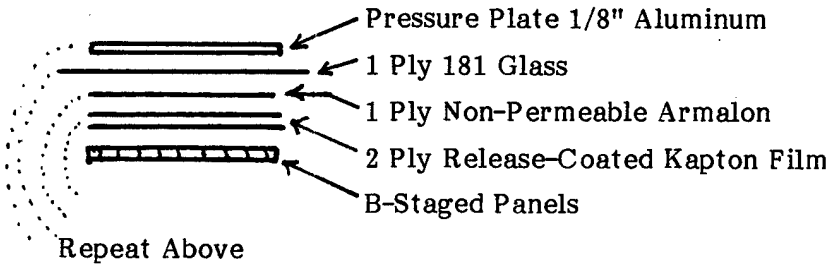
A1. Process for Graphite NTS-20 Composites

A two-step process was generally required due to the presence of solvent in the prepreg. In the first drying cycle stage, the panel was laid up and processed according to the following arrangement:



Drying Cycle: 1 hr 600°F/100 psi

In the second consolidation stage, the panel was laid up and processed according to the following arrangement:



Final consolidation: 6 hrs 650°F/200 psi

A2. Process for Graphite PPS Composites

A one-step process was employed when making graphite/PPS composites using graphite fabric and PPS film. Based on the PPS film thickness, the proper ratio of graphite fabric and PPS film was interleaved, allowing a minimum of two layers of PPS film on both the top and bottom of the stack. The layup was then envelope bagged using a release-coated Kapton film and in most cases, pressure plates. The assembly was autoclave-processed for 1 hour at 600°F under 200 psi pressure. The composite part was cooled under pressure to below 150°F and removed from the vacuum bag.

A3. Process for Graphite/PEEK Composites

A one-step process was employed to prepare graphite/PEEK composites using graphite fabric and PEEK film. The proper ratio of graphite fabric and PEEK film was stacked and envelope bagged using the same procedure described in Appendix A2. The vacuum bagged assembly was introduced into a platen press and 200 psi positive pressure was applied to the part. The assembly was heated to 750°F for 1 hour, then cooled to below 150°F before removing the composite from the vacuum bag.

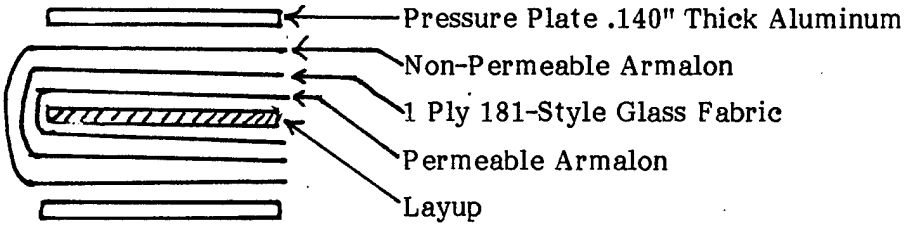
A4. Process for Graphite LaRC-2 Composites

A two-step process was used to prepare a sufficient quantity of LaRC-2/graphite fabric prepreg. In the first step, one-half of the resin was swept into the fabric using polyethylene "sweeps". The material was then dried in an air circulating oven, gradually increasing the temperatures from ambient to 250°F. In the second step, the remaining resin was applied to the previously dried prepreg. A 16 hour drying time under ambient conditions was used between resin applications. The prepreg was then dried in an air circulating oven by slowly raising temperature to 150°F and holding 15 minutes then slowly raising the temperature to 250°F and maintaining for 30 minutes.

The prepreg was stacked, and a conventional surface bleeder was applied using porous armalon release fabric between the part and the bleeder system. The part was then envelope bagged using Kapton film and sealed with a silicone sealant tape. The part was cured in an autoclave at 200 psi pressure, heating to 650°F and maintaining for 1 hour. The composite was removed from the vacuum bagged assembly after cooling under pressure to approximately 150°F.

A5. Process for Graphite Ultem Composites

A quantity of Ultem resin sufficient to provide prepreg having the desired resin content, was placed in a 30 percent methylene chloride solution and applied to graphite fabric. After 16 hours at ambient conditions the prepreg was dried in an air circulating oven for 30 minutes at 150°F, 30 minutes at 250°F and 30 minutes at 350°F. The prepreg was then stacked and envelope bagged, using the following layup.



The vacuum bag assembly containing the Ultem graphite composite was autoclave-consolidated at 600°F and 100 psi, with positive pressure applied when the assembly was at 600°F. The assembly was maintained at temperature for 60 minutes then cooled under pressure to 150°F before removing the composite from the vacuum bag.

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19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Norbornene Terminated Polysulfone NTS
Polyphenylene Sulfide (PPS)
LARC TPI (Thermoplastic Polyimide)
Polyetheretherketone (PEEK)
Ultem (Polyetherimide from G.E.)
Fluid Resistance
Kevlar (DuPont Aramid Fiber)
Graphite Fabric

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Five thermoplastic resins were screened for suitability as fluid resistant matrices for graphite composites. Additionally, the thermoplastic resins were screened for reformability after obtaining a full complement of mechanical properties. Three of these resins (NTS), (PPS) and (PEEK) were selected for further characterization. These materials were essentially insensitive to aircraft fluids and moisture and had mechanical properties equivalent to or

better than state-of-the-art (SOTA) epoxies. The PPS system was similar to NTS and PEEK with regard to room temperature properties. It was better than NTS but worse than PEEK with regard to solvent resistance. At elevated temperature (i.e. greater than 220°F) PPS was somewhat poorer than NTS and PEEK. To improve coupling between NTS and Kevlar fabric, polyimide sizings were evaluated, and yielded excellent results based on Flexural and short beam shear properties. The design properties of NTS on Kevlar and Kevlar/graphite hydrids were judged to be equivalent to those of currently used epoxy systems.